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T-111 RANKINE SYSTEM CORROSION TEST LOOP

Volume II

by R. W. Harrison, E. E. Hoffman, and J. P. Smith

Nuclear Systems Programs Missile and Space Division
GENERAL ELECTRIC COMPANY

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center
Contract NAS 3-6474

FOREWORD

The work described herein was performed by the General Electric Company, Nuclear Systems Programs (NSP), under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-6474. The primary purpose of the Advanced Refractory Alloy Corrosion Loop Program was to evaluate the compatibility of candidate refractory alloys in contact with alkali metal working fluids under conditions simulating those anticipated in projected space electric power systems. This program which was initiated in 1965 consisted of four principal investigations, namely the T-111 Rankine System Corrosion Test Loop, 1900°F Lithium Loop, Advanced Tantalum Alloy Capsule Tests, and the 2500°F Lithium Thermal Convection Loop Test. This report describes the T-111 Rankine System Corrosion Test Loop, which shall be referred to as the T-111 Corrosion Loop in this report.

The basic design of the Corrosion Test Loop was developed and proven on a prior program,* also sponsored by NASA - Lewis Research Center, and only a limited number of minor design modifications were required for the T-111 alloy system. J. Holowach was responsible for making these design modifications.

Preparation of the specifications for the purchase of the refractory alloy materials and the performance of the quality assurance testing required prior to the release of the material for fabrication were the responsibility of R. G. Frank.

W. R. Young, P. A. Blanz, and H. Mann were responsible for the fabrication of the test loop. Dr. R. B. Hand, H. Bradley, L. E. Dotson, J. Reeves, and L. A. Paian were responsible for the purification, handling, and sampling of the alkali metals used in the loop test. W. H. Bennethum made noteworthy contributions in the various phases of loop instrumentation, particularly in the areas of thermocouple calibration and installation. Dr. T. F. Lyon was responsible for the calibration of the partial pressure analyzer and the

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* NASA Contract NAS 3-2547.

interpretation of the spectra obtained during test operation. T. P. Irwin and A. C. Losekamp instrumented the loop and together with D. E. Field, S. Roof, and M. Hamilton monitored the operation of the loop during the 10,000-hour endurance test. T. Irwin and A. C. Losekamp were also responsible for the disassembly of the loop following test and the preparation of specimens for chemical, metallographic, and mechanical property evaluation and the compilation of the results of these investigations. J. P. Smith led the rather extensive posttest evaluation effort and was assisted in this work by A. Losekamp, I. Miller, and G. Anderson. The authors also wish to acknowledge the efforts of Ms. Carol Kiefel in the preparation of this report.

This program was administered for the General Electric Company by E. E. Hoffman and Dr. J. W. Semmel. R. W. Harrison acted as the Program Manager of the Advanced Refractory Alloy Corrosion Loop Program.

R. L. Davies and T. A. Moss acted as the Technical Managers for the National Aeronautics and Space Administration.

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APPENDIX A

LIST OF DRAWINGS OF THE T-111 CORROSION LOOP

GENERAL ELECTRIC

SPACE POWER & PROPULSION SECTION

LIST OF DRAWINGS

ASSEMBLY TITLE Corrosion Loop **ASSEMBLY NO.** 246R897

PROGRAM TITLE Advanced Refractory Alloy **CONTRACT NO.** NAS 3-6474

ITEM NO.	DRAWING NUMBER	DWG LEVEL 1	2	3	4	DESCRIPTION/TITLE	REV.	ISSUE DATE	CODE
	263E807G1	1				Surge Tank Ass'y - Li	A	4-29-67	
	941D864	1	1			Tube	B	5-2-67	
	941D881G1	1	2			EM Pump Duct Ass'y		4-14-66	
	142B1946P1	1	2	1		Cap, Duct - Li Pump		4-14-66	
	142B1948P1	1	2	2		Pipe, Wrapper Li Pump		4-14-66	
	119C2990G1	1	2	3		Finned Duct		4-14-66	
	142B1949P1	1	2	3	1	Duct Li Pump		4-14-66	
	142B1947P1	1	2	3	2	Duct Cap - Li Pump		4-14-66	
	142B1945P1	1	2	4		Connector - Li Pump		4-14-66	
	142B1373	1	2	5		Torsion Tube		4-14-66	
	941D864	1	1	X		Tube		4-14-66	
	142B1924P1	1	3			Reducer		4-14-66	
	142B1925P1	1	4			Thermocouple Well		4-14-66	
	941D753	1	5			Fitting	B	4-14-66	
	119C2973P1	1	6			Coil		4-14-66	
	47C140159G1	1	7			Bi-Metallic Joint		4-14-66	
	142B1920P1	1	8			Tube	A	4-29-67	
	941D869G1	1	9			Surge Tank		4-14-66	
	263E82[illegible]G1	2				Li Heater Line Ass'y	A	5-1-67	
	941D864	1	1			Tube		4-14-66	
	941D883G1	2	2			Heater Ass'y		4-14-66	
	119C2997	2	2	1		Electrode		4-14-66	
	142B1953	2	2	2		Coil		4-14-66	
	263E828G1	3				Boiler Ass'y - K		4-14-66	
	941D864	1	1			Tube		4-14-66	
	941D884G1	3	1			Pre-Heater Ass'y		4-14-66	
	119C2997	2	2	1		Electrode		4-14-66	
	142B1954	3	1	1		Coil Pre-Heater	B	11-11-66	
	142B1955P1	3	1	2		Insert, Thermocouple		4-14-66	

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ASSEMBLY TITLE Corrosion Loop ASSEMBLY NO. 246R897

PROGRAM TITLE Advanced Refractory Alloy CONTRACT NO. NAS 3-6474

ITEM NO.	DRAWING NUMBER	DWG LEVEL 1	2	3	4	DESCRIPTION/TITLE	REV.	ISSUE DATE	CODE
	263E809G1	3	2			Boiler Ass'y		4-14-66	
	941D864	1	1	X		Tube		4-14-66	
	119C2979G1	3	3			Turbine Simulator, 1 Stage		4-14-66	
	119C2978	3	3	1		Reducer		4-14-66	
	119C2977G1	3	3	2		Nozzle Ass'y		4-14-66	
	119C2976	3	3	2	1	Blade Ass'y		4-14-66	
	119C2975	3	3	2	2	Nozzle Ass'y		4-14-66	
	142B1927P1	3	3	3		Casing		4-14-66	
	142B1931P1	3	3	4		Thermocouple Well		4-14-66	
	142B1930P1	3	3	5		Transducer Connector		4-14-66	
	941D864	1	1	X		Tube		4-14-66	
	142B1930P2	3	3	5		Transducer Connector		4-14-66	
	263E813G1	4				Pump Outlet Ass'y - K		5-3-66	
	263E827G1	4	1			Valve, Modified Hoke (Metering)	A	5-27-66	
	119C2893G1	4	1	1		Ball Bearing Stem Ass'y	D	5-27-66	
	47C140161G1	4	1	2		Yoke, Modified	B	11-1-66	
	142B1974P1	4	1	2	1	Pinion Mount	A	5-27-66	
	119C2624P1	4	1	3		Spur Gear	B	11-1-66	
	119C2625P1	4	1	4		Pinion, Involute	B	11-1-66	
	142B1815P1	4	1	5		Bushing, Modified	C	5-27-66	
	142B1859P1	4	1	6		Collar		12-11-64	
	941D879P1	4	2			Tube		4-14-66	
	941D753P5	1	5			Tee	B	4-14-66	
	263E808G1	5				Surge Tank Ass'y - K	A	5-1-67	
	263E643P1	5	1			EM Pump, Modified	A	5-1-67	
	941D864	1	1			Tube		4-14-66	
	941D753	1	5			Fitting	B	4-14-66	
	119C2969P1	5	2			Isolation Valve, Modified		4-14-66	
	263E827G2	4	1	X		Valve, Modified Hoke (Isolation)	A	5-27-66	

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LIST OF DRAWINGS

ASSEMBLY TITLE Corrosion Loop **ASSEMBLY NO** 263E897

PROGRAM TITLE Advanced Refractory Alloy **CONTRACT NO.** NAS 3-6474

ITEM NO.	DRAWING NUMBER	DWG LEVEL 1	2	3	4	DESCRIPTION/TITLE	REV.	ISSUE DATE	CODE
	119C2893G1	4	1	1	X	Ball Stem Ass'y	D	5-27-66	
	47C140161G1	4	1	2	X	Yoke	A	5-27-66	
	142B1974P1	4	1	2	1	X Pinion Mount	A	5-27-66	
	119C2624P1	4	1	3	X	Spur Gear	A	5-27-66	
	119C2625P1	4	1	4	X	Pinion	A	5-27-66	
	142B1815P1	4	1	5	X	Bushing - Modified	C	5-27-66	
	142B1859P1	4	1	6	X	Collar	B	5-27-66	
	47C140159G1	1	7			Bi-Metallic Joint		4-14-66	
	119C2970P1	5	3			Coil - Argon Press - K		4-14-66	
	941D897G1	6				Turbine Simulator - Condenser	A	4-29-67	
	941D864	1	1			Tube		4-14-66	
	142B1351P1	6	1			Bracket		4-14-66	
	941D874G1	6	2			Turbine Simulator - Nine Stage	A	4-29-67	
	119C2977	3	3	2		Nozzle Ass'y		4-14-66	
	119C2976	3	3	2	1	Blade Ass'y		4-14-66	
	119C2975	3	3	2	2	Nozzle		4-14-66	
	142B1932P1	6	2	1		Key		4-14-66	
	142B1929P1	6	2	2		Transducer Connector		4-14-66	
	142B1926P1	6	2	3		Reducer		4-14-66	
	142B1928P1	6	2	4		Casing		4-14-66	
	119C2978P1	3	3	1		Reducer		4-14-66	
	142B1931P1	3	3	4		Thermocouple Well		4-14-66	
	941D864	1	1	X		Tube		4-14-66	
	263E825G1	6	3			Condenser		4-14-66	
	941D894G1	6	4			Sub-Cooler Reservoir		4-14-66	
	941D882P1	7				Tube, Pump Return - Li		4-14-66	
	263E818G1	8				Spool Ass'y		4-14-66	
	142B1365P1	8	2			Mounting Bracket		4-14-66	
	243R613G1	9				Support Structure		4-14-66	

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LIST OF DRAWINGS

ASSEMBLY TITLE Corrosion Loop ASSEMBLY NO. 246R897

PROGRAM TITLE Advanced Refractory Alloy CONTRACT NO. NAS 3-6474

ITEM NO.	DRAWING NUMBER	DWG LEVEL 1	2	3	4	DESCRIPTION/TITLE	REV.	ISSUE DATE	CODE
	263E754G1	10				Shield Drive Ass'y	A	12-14-64	
	142B1834P1	10	1			Race Mount		11-18-64	
	142B1833P1	10	2			Outer Race		11-18-64	
	142B1832P1	10	3			Inner Race		11-18-64	
	142B1797P1	10	4			Washer	A	11-18-64	
	119C2760G1	10	5			Condenser Shield - Rotating	B	1-4-65	
	142B1831G1	10	6			Rack Drive Ass'y	A	2-4-65	
	142B1826P1	10	6	1		Drive Screw		11-18-64	
	142B1827G1	10	6	2		Bracket	B	2-4-65	
	142B1825P1	10	7			Thrust Bearing		11-18-64	
	941D787G1	10	8			Base	C	6-21-65	
	142B1830P1	10	9			Rack Support		11-18-64	
	119C2913P1	10	10			Rack		11-18-64	
	119C2921P1	10	11			Rod - Spacer		12-17-64	
	142B1829P1	10	12			Rack Support		11-18-64	
	142B1828P1	10	13			Clamp		11-18-64	
	941D794P1	10	14			Support Plate - Upper	B	6-21-65	
	119C2995G1	11				Pressure Metering System	A	5-1-67	
	47C140160G1	11	1			Bi-Metallic Joint	A	10-3-66	
	263E826G1	12				Press Metering System	A	4-29-67	
	47C140160G1	11	1			Bi-Metallic Joint	A	10-3-66	
	941D896G1	13				Pressure Metering System		4-14-66	
	47C140160G1	11	1			Bi-Metallic Joint	A	10-3-66	
	941D892	14				Bus Bar		4-14-66	
	142B1754	15				Spacer		7-21-64	
	941D893	16				Bus Bar		4-14-66	
	142B1761P1	17				Insulator		8-28-64	
	119C2870G1	18				Cable Ass y		7-21-64	
	142B1649	18	1			Connector		4-21-64	

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LIST OF DRAWINGS

ASSEMBLY TITLE Corrosion Loop ASSEMBLY NO. 246R897

PROGRAM TITLE Advanced Refractory Alloy CONTRACT NO. NAS 3-6474

ITEM NO.	DRAWING NUMBER	DWG LEVEL 1	2	3	4	DESCRIPTION/TITLE	REV.	ISSUE DATE	CODE
	142B1650	18	2			Connector		4-21-64	
	119C2873	19				Cable Ass'y		7-21-64	
	142B1649	18	1			Connector		4-21-64	
	142B1650	18	2			Connector		4-21-64	
	SK56131-326	20				Electric Feedthru		12-8-63	
	142B1782	21				Electric Feedthru	A	12-17-64	
	SK56131-372	22				Feedthru	B	4-14-66	
	263E760G1	23				Ass'y Shield Boiler Radiation		2-10-65	
	941D801G1	23	1			Ass'y Inner Cover		2-10-65	
	941D798G1	23	2			Ass'y Cover	A	2-10-65	
	941D797G1	23	3			Spider Cover	A	2-10-65	
	941D800	23	4			Ass'y Outer Cover	A	6-21-65	
	119C2926	23	5			Plate Bottom		2-10-65	
	119C2927	23	6			Shield Heat Radiation		2-10-65	
	142B1861	23	7			Rod Support	B	6-21-65	
	142B1862	23	8			Clevis	A	2-10-65	
	142B1860	23	9			Support		2-10-65	
	119C2992G1	24				Pressure Transducer Stressed Dia.		4-14-66	
	142B1839P1	24	1			LVDT Coil	B	6-7-66	
	119C2993P1	24	2			Cap		4-14-66	
	119C2991G1	24	3			Body Ass'y		4-14-66	
	142B1863G2	24	3	1		Diaphragm Ass'y	A	4-14-66	
	165A5257P1	24	3	1	1	Diaphragm		4-14-66	
	142B1371P1	24	3	2		Cap		4-14-66	
	142B1370P1	24	3	3		Housing		4-14-66	
	142B1952P1	24	3	4		Body		4-14-66	
	142B1951P1	24	3	5		Reducer		4-14-66	
	941D864	1	1	X		Tube		4-14-66	
	142B1350	25				Stud		4-14-66	

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SPACE POWER & PROPULSION SECTION

LIST OF DRAWINGS

ASSEMBLY TITLE Corrosion Loop ASSEMBLY NO. 246R897

PROGRAM TITLE Advanced Refractory Alloy CONTRACT NO. NAS 3-6474

ITEM NO.	DRAWING NUMBER	DWG LEVEL 1	2	3	4	DESCRIPTION/TITLE	REV.	ISSUE DATE	CODE
	142B1367	26				Hex Nut - Special		4-14-66	
	142B1368	27				Flat Washer - Special		4-14-66	
	142B1374	28				Lugs, Torsion Tube, EM Sump		4-14-66	

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REPLACES 6-2-66

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APPENDIX B

MAJOR DRAWINGS OF THE T-111 RANKINE SYSTEM CORROSION TEST LOOP

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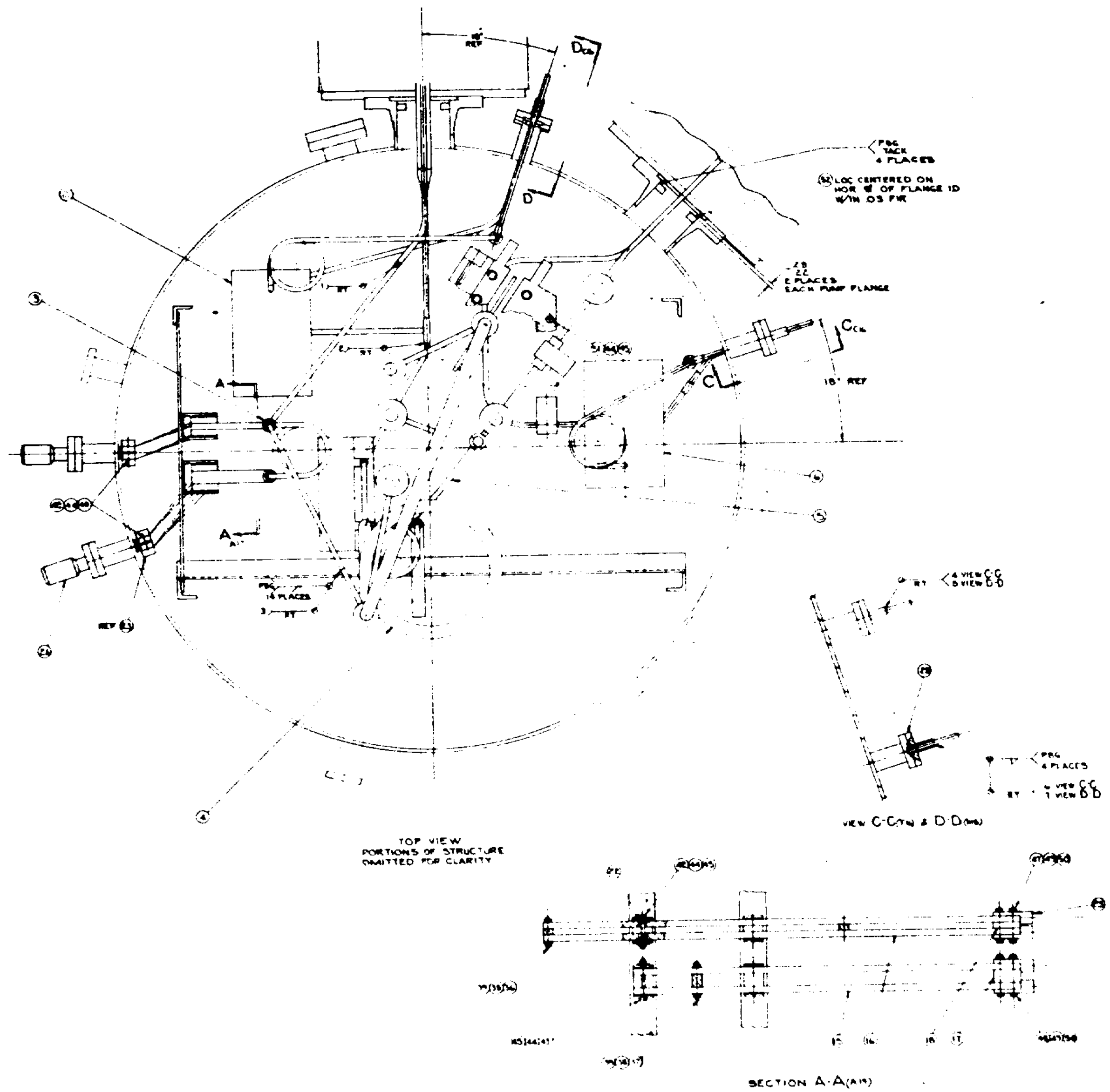

Figure B-1. T-111 Rankine System Corrosion Test Loop Assembly (Drawing No. 246R897).

Figure B-1. Continued.

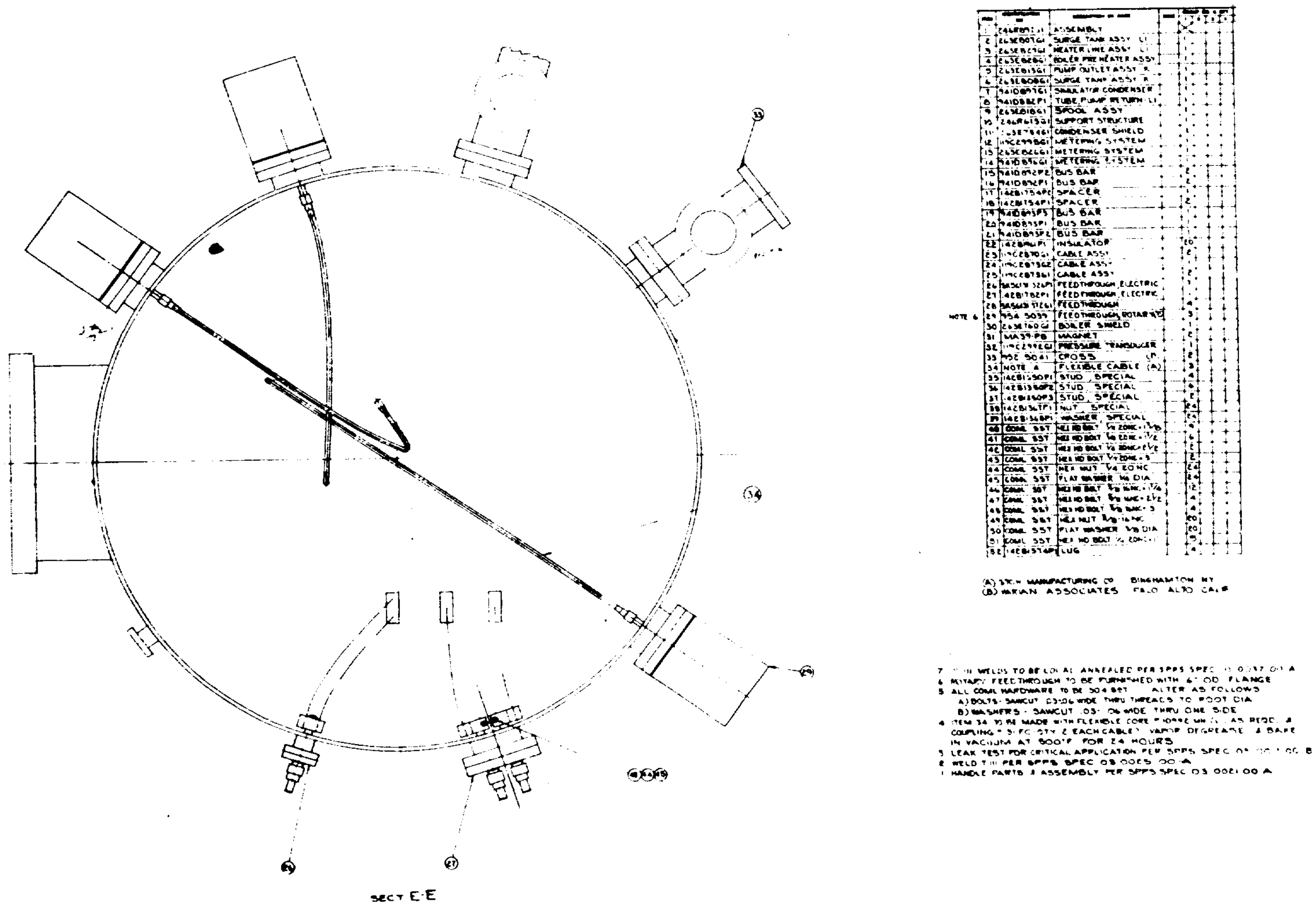

Figure B-1. Concluded.

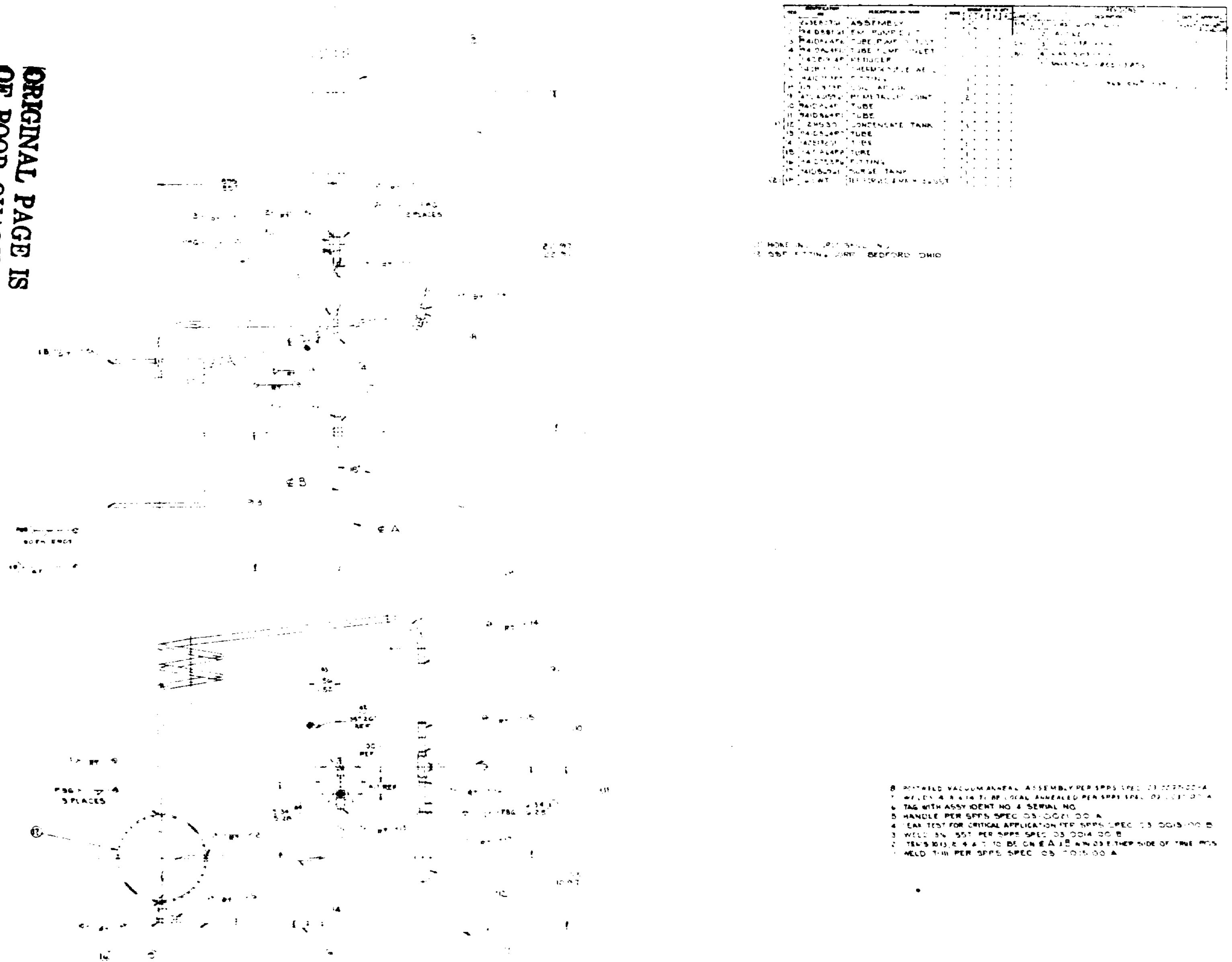

Figure B-2. Lithium Surge Tank Assembly (Drawing No. 263E807 (Rev.A)).

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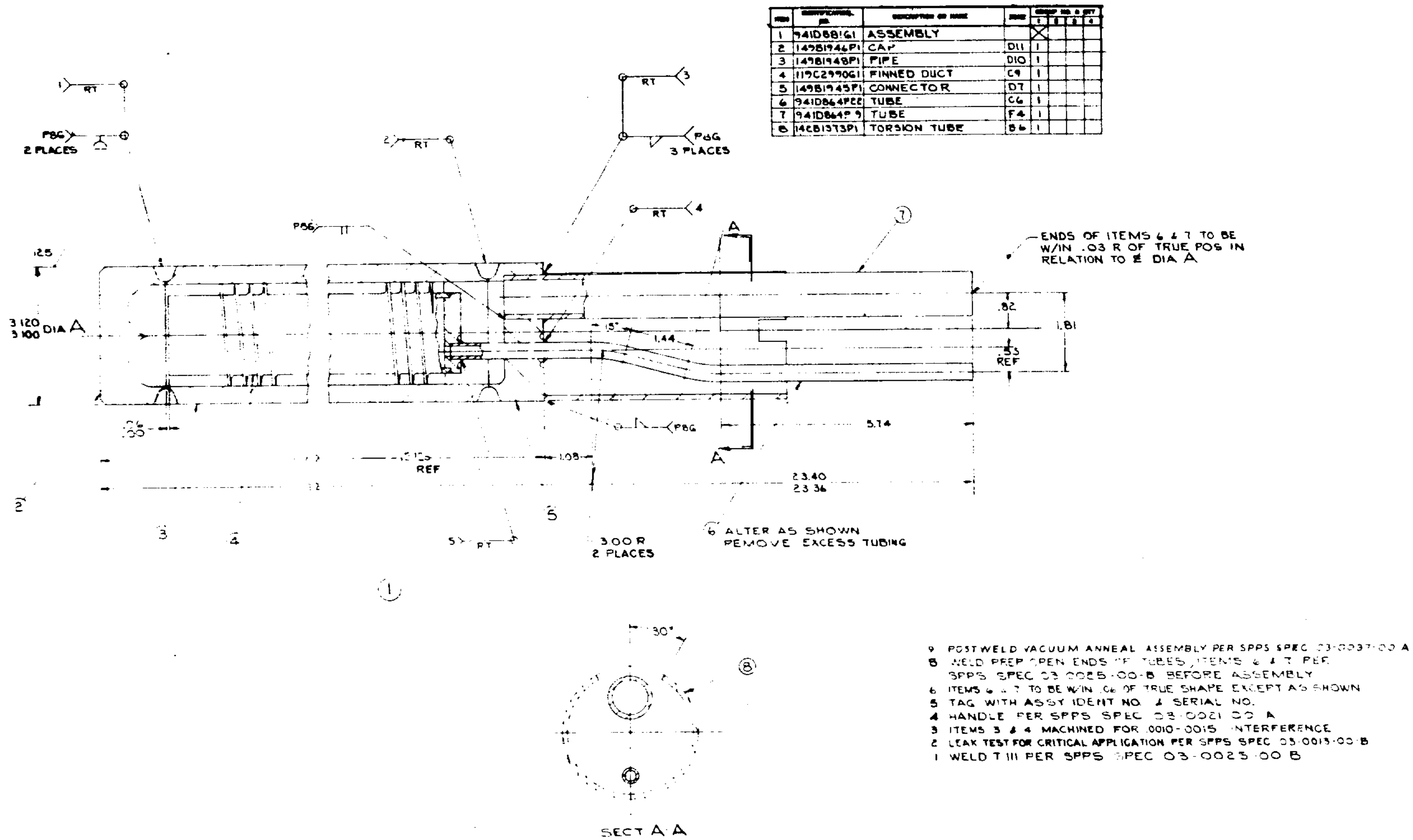


Figure B-3. Lithium EM Pump Duct Assembly (Drawing No. 941D881).

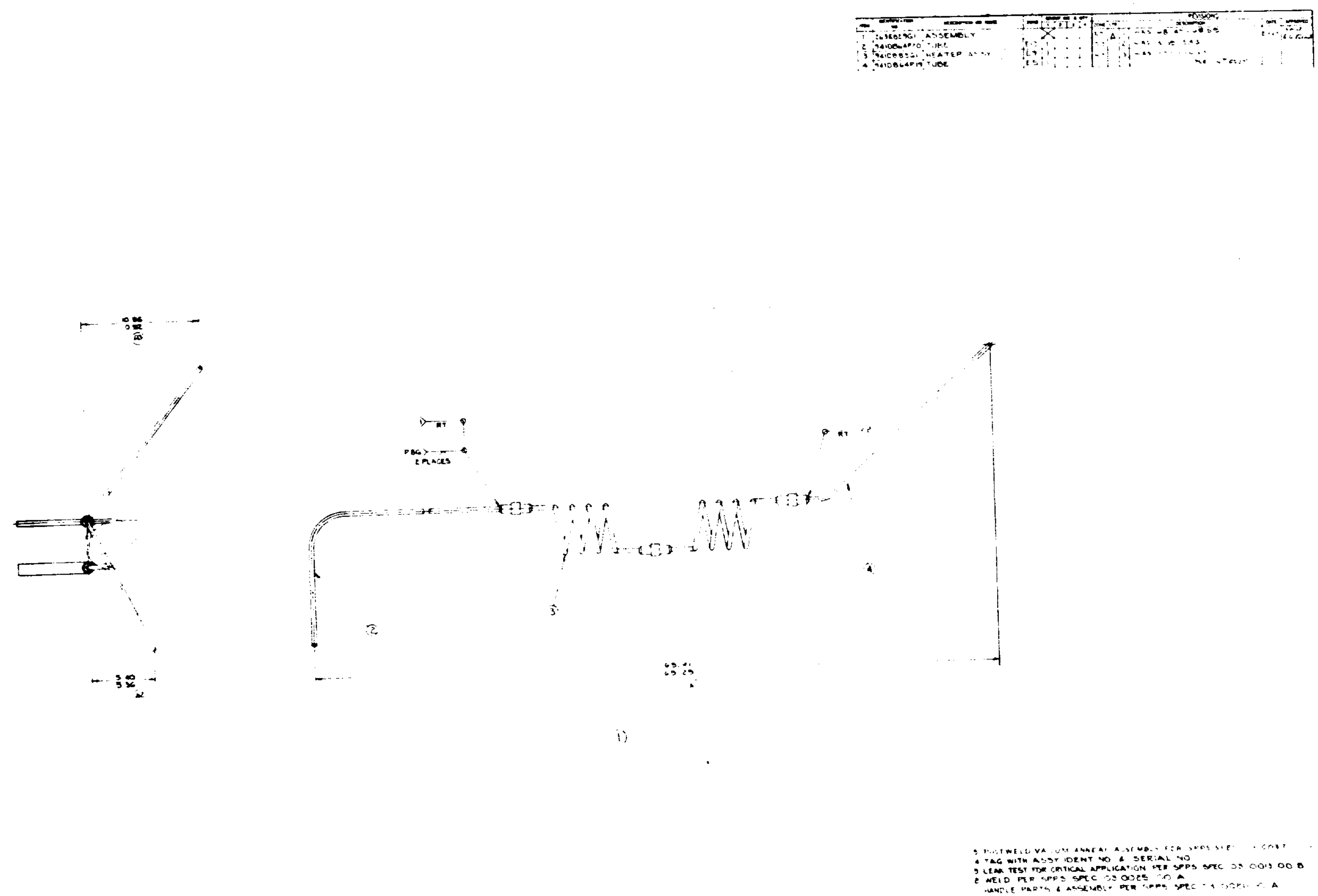
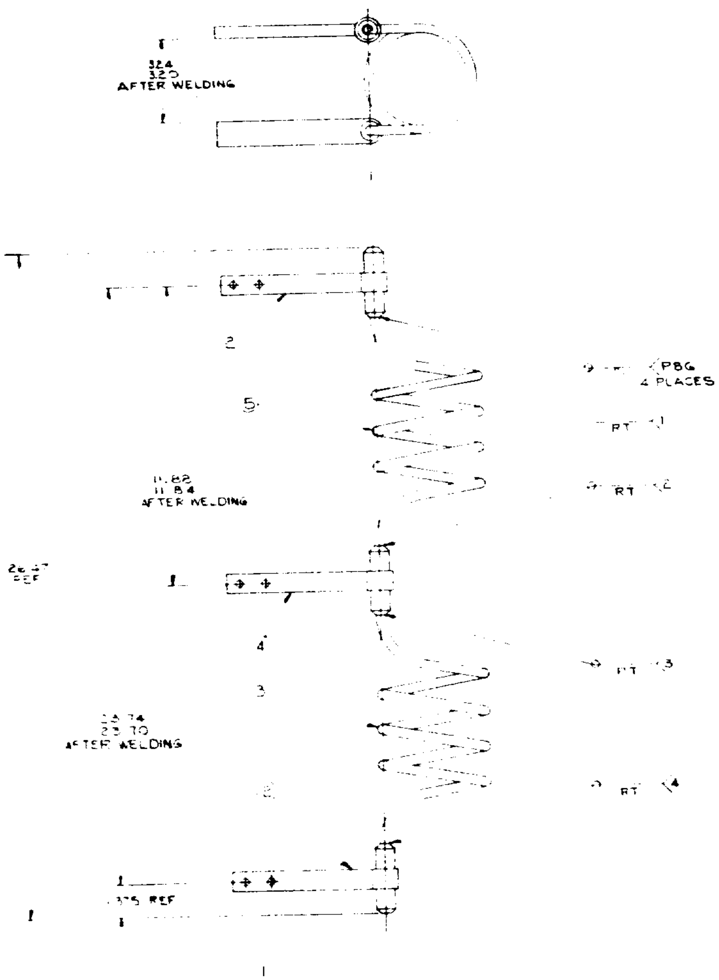


Figure B-4. Lithium Heater Line (Drawing No. 263E829 (Rev. A).



ITEM	IDENTIFICATION NO	DESCRIPTION OR NAME	ZONE	GROUP NO & QTY 1	2	3	4
1	941D883G1	ASSEMBLY		X			
2	119C2997G1	ELECTRODE	B9	2			
3	142B1953P1	COIL	C9	1			
4	119C2997G2	ELECTRODE	C9	1			
5	142B1953P2	COIL	C9	1			

4 TAG WITH ASSY IDENT NO. & [illegible]ERIAL NO.
3 LEAK TEST FOR CRITICAL APPLICA[illegible]ON PER SPPS SPEC 03-0013-00-B
2 WELD PER SPPS SPEC 03-[illegible].5-00-A
1 HANDLE PER SPPS SPEC 0[illegible] 3021-00-A

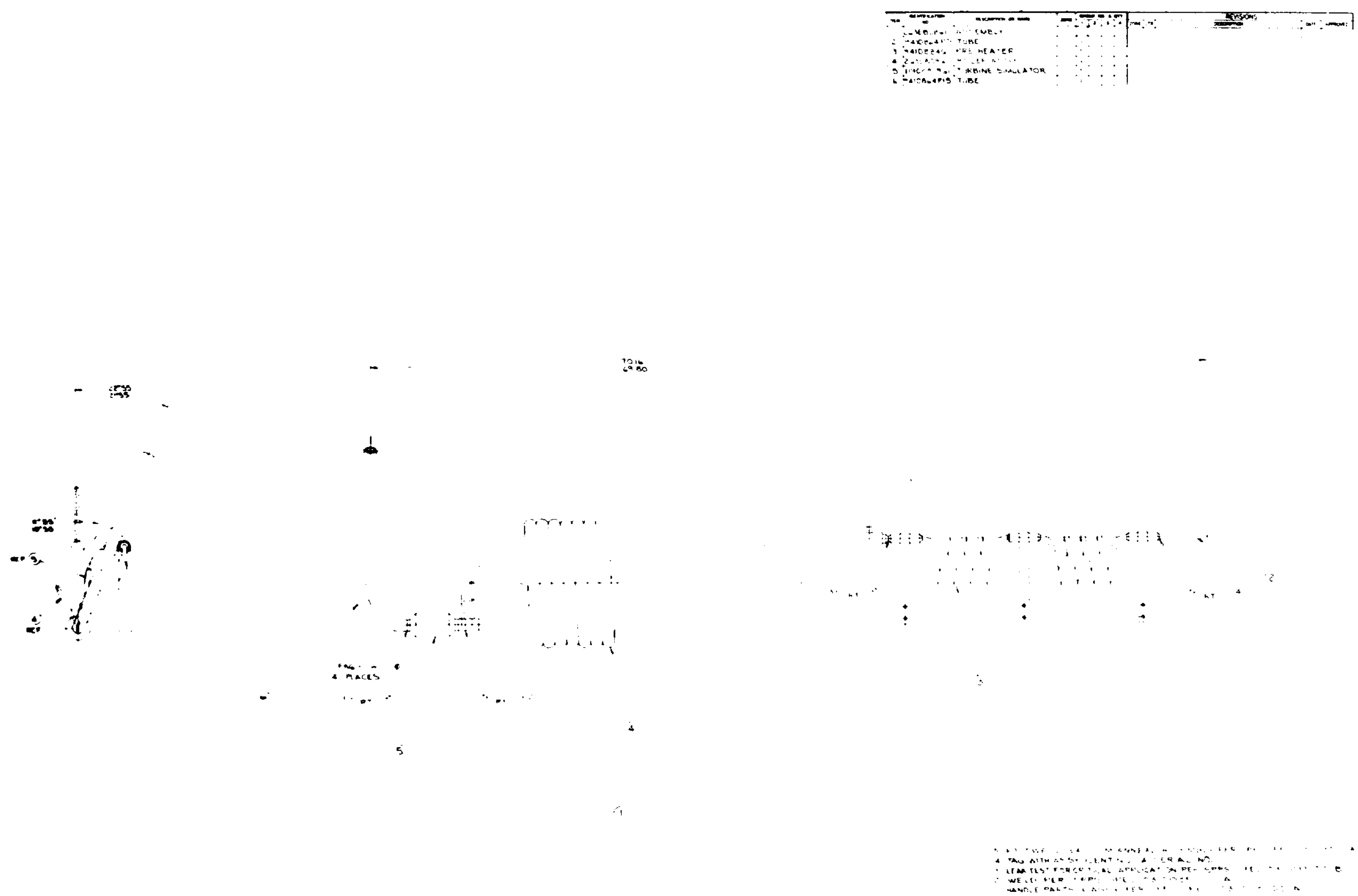
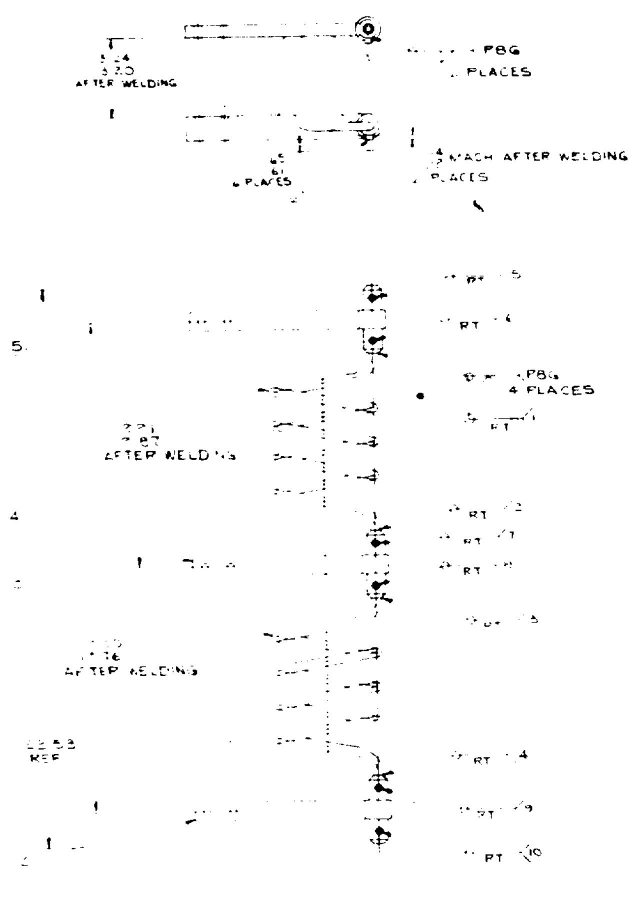


Figure B-6. Potassium Boiler Assembly (Drawing No. 263E828).

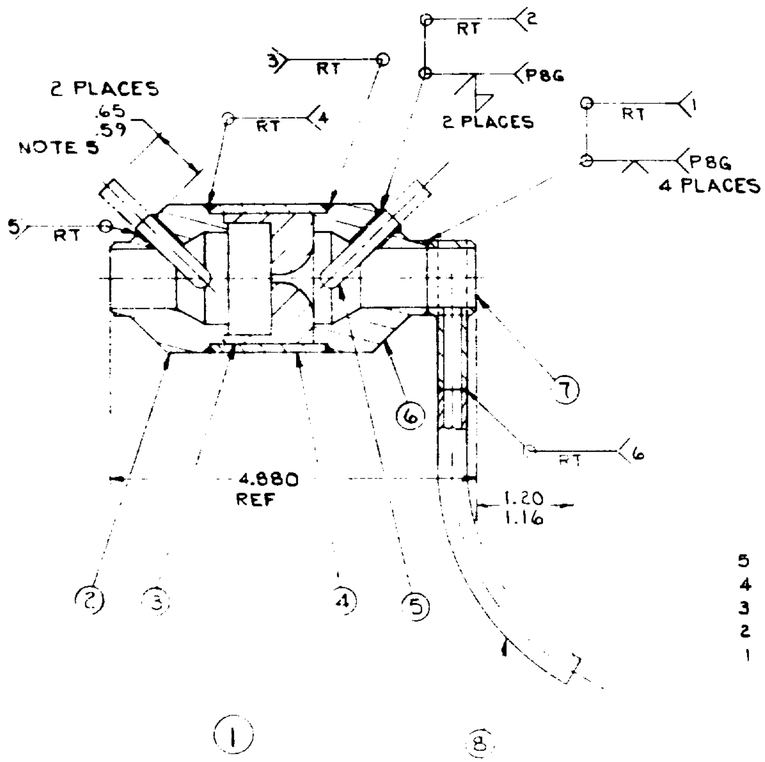


ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP NO. & QTY 1	2	3	4
1	941D884G1	ASSEMBLY		X			
2	119C2997G3	ELECTRODE		2			
3	142B1954P1	COIL		1			
4	119C2997G4	ELECTRODE		1			
5	142B1954P2	COIL		1			
6	142B1955P1	INSERT		6			

4 TAG WITH ASSY IDENT NO. & SERIAL NO
3 LEAK TEST FOR CRITICAL APPLICATION PER SPPS SPEC 03-0013-00-5
2 WELD PER SPPS SPEC 03-0025-00-A
1 HANDLE PER SPPS SPEC 03-0021-00-A

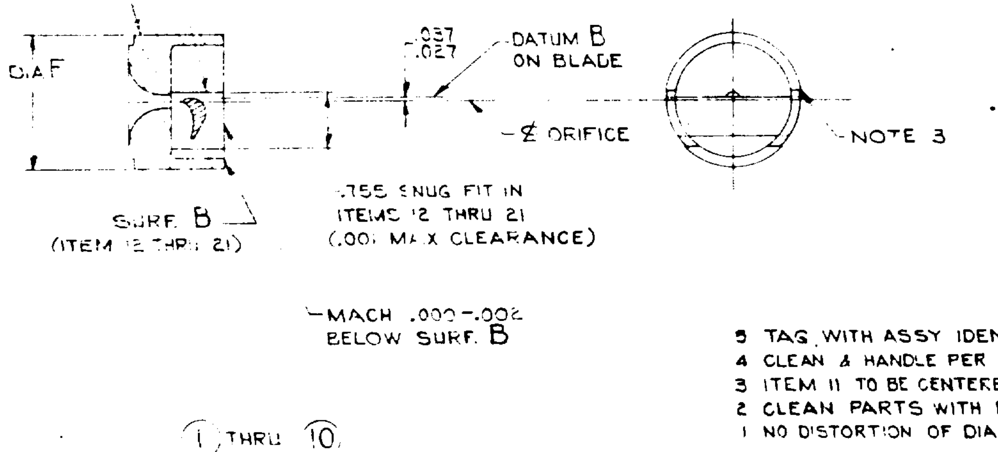
Figure B-7. Preheater Assembly (Drawing No. 941D884)

ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP NO. & QTY 1	2	3	4
1	119C2979G1	ASSEMBLY		X			
2	119C2978P2	REDUCER		1			
3	119C2977G1	NOZZLE ASSY		1			
4	142B1927P1	CASING		1			
5	142B1931P1	THERMOCOUPLE WELL		2			
6	119C2978P1	REDUCER		1			
7	142B1930P1	TRANSDUCER CONNECTOR		1			
8	941D864P17	TUBE		1			



5 AFTER ASSY CUT OFF ITEM 5 FLUSH WITH WELD
4 TAG WITH ASSY IDENT NO. & SERIAL NO.
3 HANDLE PER SPPS SPEC 03-0021-00-A
2 LEAK TEST FOR CRITICAL APPLICATION PER SPPS SPEC 03-0013-00-B
1 WELD T-111 PER SPPS SPEC 03-0025-00-A

ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP No. & QTY 1	2	3	4	5	6	7	8	9	10
1	119C2977 G1	ASSEMBLY											
2	G2												
3	G3												
4	G4												
5	G5												
6	G6												
7	G7												
8	G8												
9	G9												
10	G10												
11	119C2976 G1	BLADE ASSEMBLY		1	1	1	1	1	1	1	1	1	1
12	119C2975 P1	NOZZLE		1									
13	P2				1								
14	P3					1							
15	P4						1						
16	P5							1					
17	P6								1				
18	P7									1			
19	P8										1		
20	P9											1	
21	P10												1



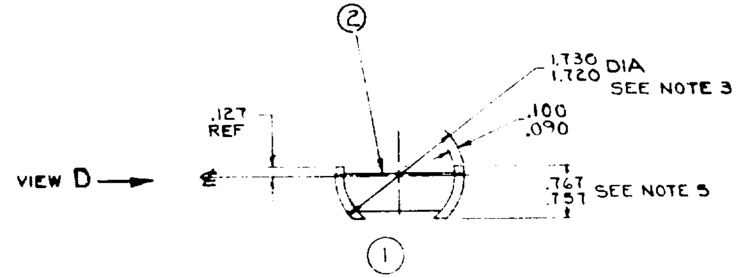
5 TAG WITH ASSY IDENT NO. & SERIAL NO.
4 CLEAN & HANDLE PER SPPS SPEC 03-0021-00-A
3 ITEM 11 TO BE CENTERED IN RELATION TO DIA F W/IN .010 FIR
2 CLEAN PARTS WITH ETHYL ALCOHOL BEFORE ASSY
1 NO DISTORTION OF DIA F ALLOWABLE AFTER ASSY

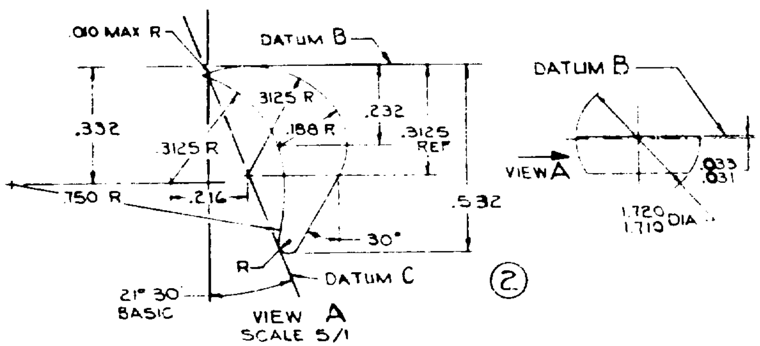
Figure B-9. Nozzle Assembly (Drawing No. 119C2977).

ITEM	IDENTIFICATION NO	DESCRIPTION OR NAME	ZONE	GROUP NO & QTY 1	2	3	4
1	119C2976 G1	ASSEMBLY		X			
2	P2	BLADE		1			
3	P3	BLOCK		1			
4	P4	BLOCK		1			

REVISIONS

SYM	ZONE	DESCRIPTION	DATE	APPROVED
A	C4	.031-.033 WAS .31-.33 PER CN # 0433		





16 SURF F ON ITEMS 3 & 4 TO BE IN SAME PLANE & SQ WITH SURF E W/IN .0005 FIR
15 SURF E ON ITEMS 3 & 4 TO BE IN SAME PLANE W/IN .0005 FIR
14 HANDLE PARTS & ASSY PER SPPS SPEC 03-0021-00-A
13 CLEAN ALL PARTS WITH ETHYL ALCOHOL
12 ALL POLISHING TO BE DONE WITH ALUMINA GRIT CLOTH
11 ITEM 2 TO HAVE .002 MAX TWIST BETWEEN ENDS
10 CONTOUR DIMENSIONS APPLY TO ITEM 2
9 CONTOUR TO BE W/IN .002 OF TRUE SHAPE EXCEPT AREA 1/4 FROM ENDS MAY HAVE ADDITIONAL .005 STOCK REMOVAL AT ASSY
8 CONTOURED SURFACES TO BE 16√ OR BETTER
7 SLOT IN ITEM 3 & 4 TO BE SNUG FIT FOR ITEM 2
6 MAX .010 LOCALIZED GAP BETWEEN SNUG FIT CONTOURS
5 PROCESS DIM., SEE NEXT ASSY FOR FINISH MACH DIM
4 Mo TZC PER SPPS SPEC 01-0011-01-C
3 ITEM 2 NOT TO EXTEND OUTSIDE OF ITEMS 3 & 4
2 CONTOUR OF ITEM 2 TO BE ELECTRO POLISHED WITH A MAX MATL REMOVAL OF .0005
1 FLUORESCENT PENETRANT INSP ITEM 2 PER ASTM-E165-63 NO DEFECTS INTERPRETED AS CRACKS ALLOWED

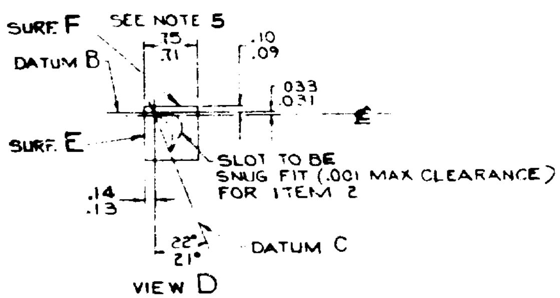
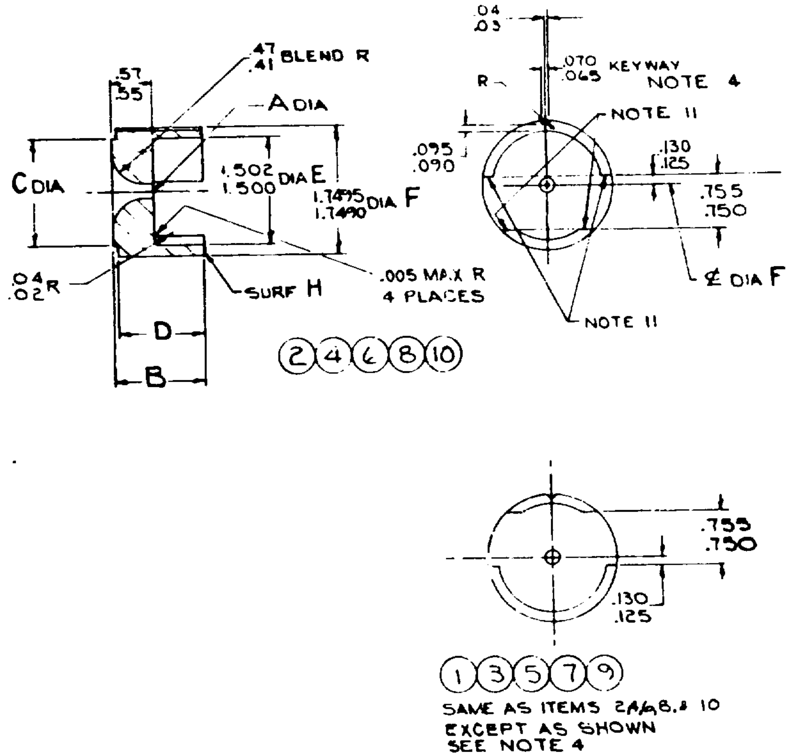


Figure B-10. Turbine Simulator Blade Assembly (Drawing No. 119C2976 (Rev. A)).



ITEM	A DIA	B DIM	C DIA	D DIM
1	.0885-.0895	1.249-1.250	—	—
2	.0875-.0885	1.249-1.250	—	—
3	.0965-.0975	1.24-1.26	1.495-1.497	1.180-1.181
4	.1065-.1075			
5	.1175-.1185			
6	.1295-.1305			
7	.1445-.1455			
8	.1595-.1605			
9	.1775-.1785			
10	.1985-.1995	1.24-1.26	1.495-1.497	1.180-1.181

11 SURF TO BE IN SAME PLANE W/IN .0005 (2 PLACES)
10 TAG WITH PART IDENT NO. & SERIAL NO.
9 FLUORESCENT PENETRANT INSP PER ASTM-E165-63. NO DEFECTS INTERPRETED AS CRACKS ALLOWED
8 ROUGH POLISHING TO BE DONE WITH ALUMINA GRIT CLOTH
7 CLEAN & HANDLE PER SPPS SPEC 03-0021-00-A
6 DIA C & E TO BE CONC WITH DIA F W/IN .002 FIR
5 DIA A TO BE CONC WITH DIA F W/IN .005 FIR
4 KEYWAY TO BE OMITTED FOR ITEM 1
3 SURF H TO BE SQ WITH DIA F W/IN .001 FIR
2 KEYWAY TO BE PARALLEL WITH DIA F W/IN .001 FIR
1 Mo-TZC PER SPPS SPEC 01-0011-01-C

Figure B-11. Turbine Simulator Nozzle (Drawing No. 119C2975).

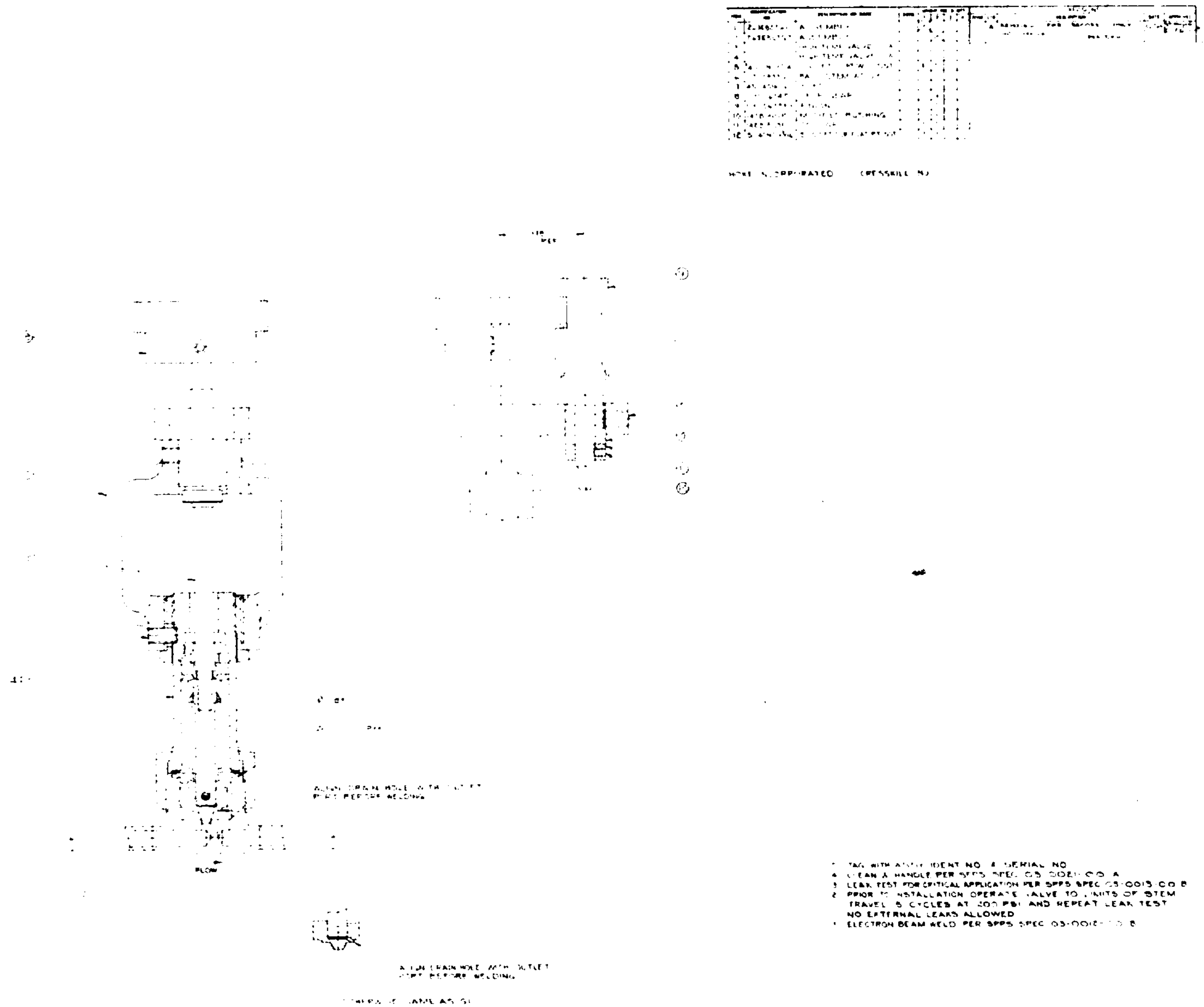


Figure B-12. High-Temperature Liquid Metal, Modified, Hoke Valve (Drawing No. 263E827 (Rev. A).

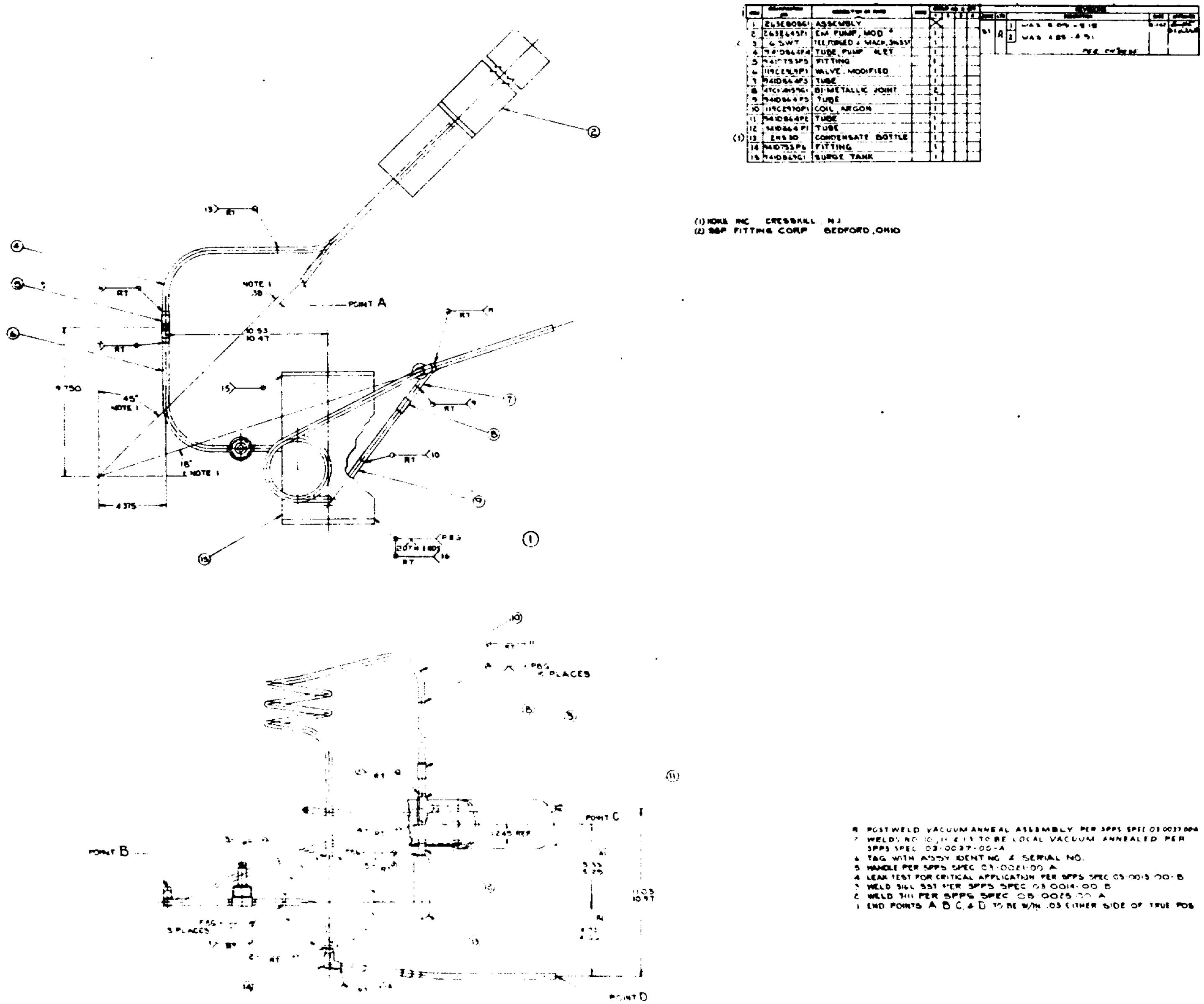


Figure B-13. Potassium Surge Tank Assembly (Drawing No. 263E808 (Rev. A).

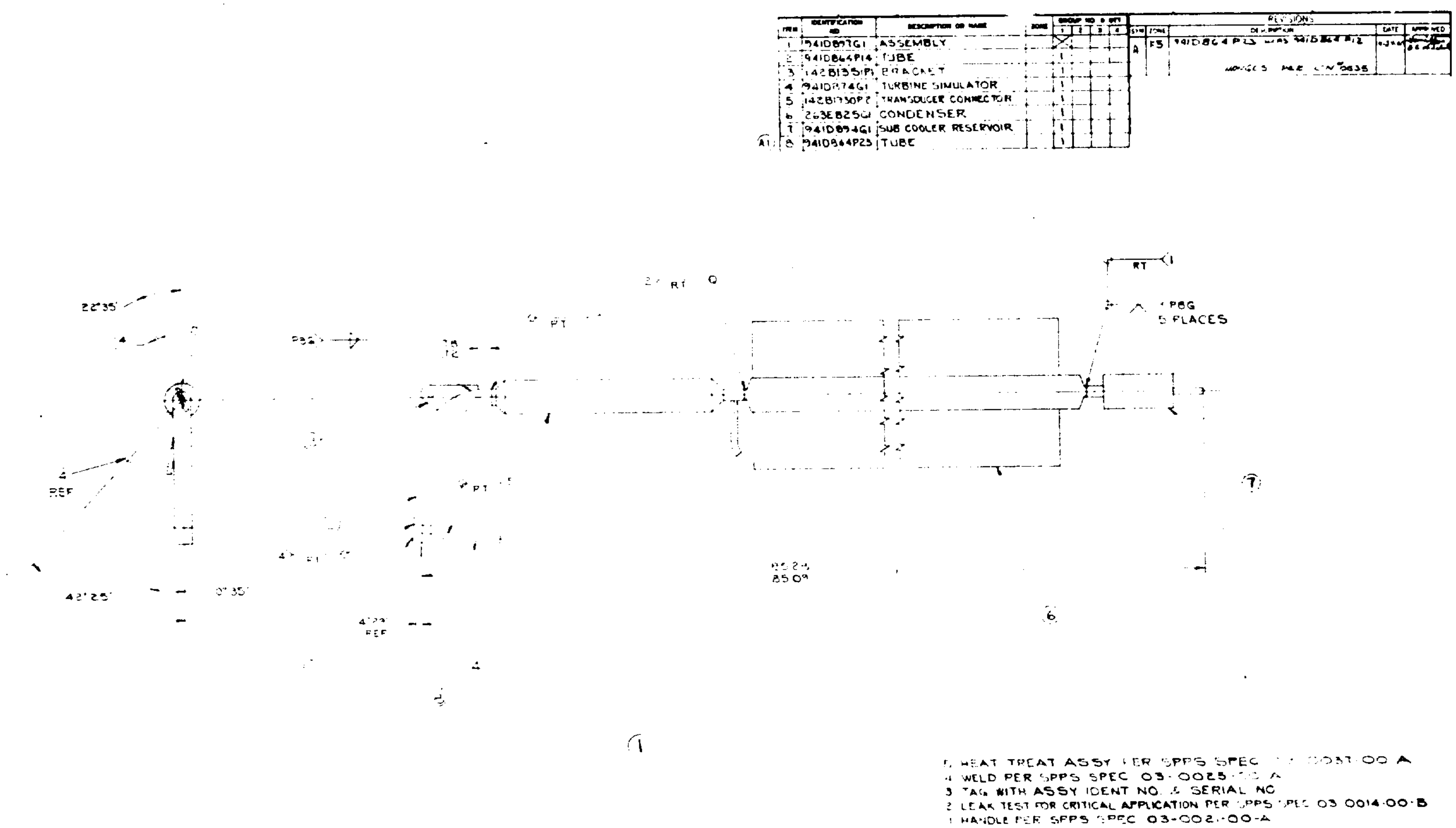
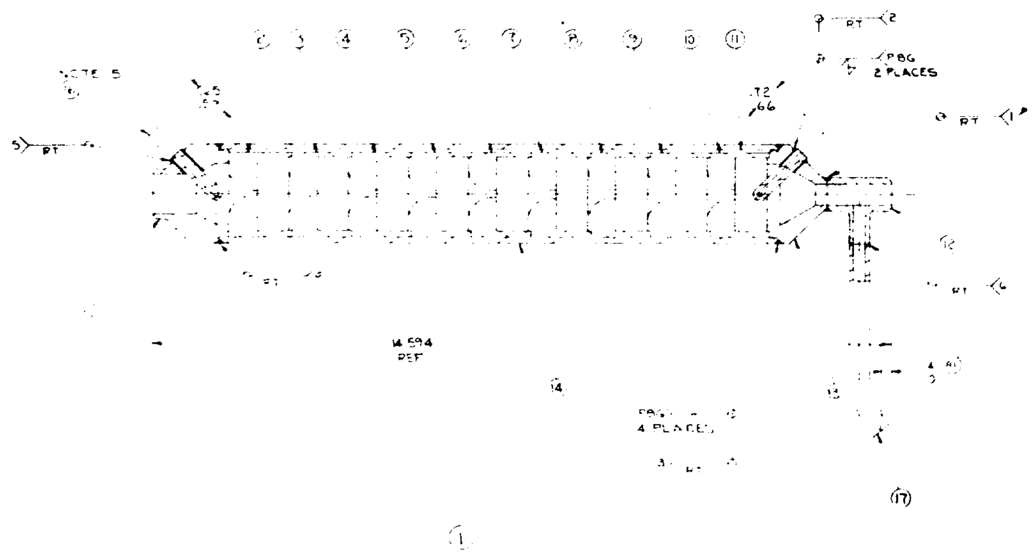


Figure B-14. Turbine Simulator and Condenser Assembly (Drawing No. 941D897 (Rev. A)).

ITEM	IDENTIFICATION NO	DESCRIPTION OR NAME	ZONE	GROUP NO & QTY 1	2	3	4
1	941D874G1	ASSEMBLY					
2	119C2977G2	NOZZLE ASSEMBLY		1			
3	G3			1			
4	G4			1			
5	G5			1			
6	G6			1			
7	G7			1			
8	G8			1			
9	G9			1			
10	119C2977G10	NOZZLE ASSEMBLY		1			
11	142B1932P1	KEY		1			
12	142B1929P1	TRANSDUCER CONNECTOR		1			
13	142B1926P1	REDUCER		1			
14	142B1928P1	CASING		1			
15	119C2978P1	REDUCER		1			
16	142B1931P1	THERMOCOUPLE WELL		2			
17	941D864P16	TUBE		1			



5 AFTER ASSY CUT OFF ITEM 16 FLUSH WITH WELD
4 TAG WITH ASSY IDENT NO & SERIAL NO
3 HANDLE PARTS & ASSY PER SPPS SPEC 03-0021-00-A
2 LEAK TEST FOR CRITICAL APPLICATION PER SPPS SPEC 03-0013-00-B
1 WELD TIII PER SPPS SPEC 03-0025-00-A

Figure B-15. Nine-Stage Turbine Simulator (Drawing No. 941D874 (Rev. A)).

A P P E N D I X   C

TYPICAL T-111 ALLOY SPECIFICATION: SPECIFICATION NO. B50YA325,
"SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY"

GENERAL ELECTRIC Nuclear Systems Programs Cincinnati, Ohio 45215	SPECIFICATION NO. B50YA325-S1
ENGINEERING SPECIFICATION	ISSUE DATE April 21, 1971

TITLE SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY	ORIGINAL CONTRACT
	SECURITY CLASSIFICATION

Prepared by: R.A. Ekvall Date 4/21/71

Approved by: W.R. Young Date 4/23/71

Approved by: ______ Date ______

Issued by: W. Wiliog 4/23/71 DRAFTING Mail Zone 26 Phone 5224/5514

SUPERSEDES SPECIFICATION NO.	DATED	REVISION AUTHORIZATION	TYPE REVISION SYSTEM
B50YA325	1-7-71		☐ CN ☐ CID

NS 1080

SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY	DATE April 21,1971	NO. B50YA325

1. SCOPE

1.1. Scope. This specification covers T-111 alloy in tube and pipe form intended for high-temperature structural application and alkali metal containment.

2. APPLICABLE DOCUMENTS

The latest revisions of the following documents apply.

2.1. Government Documents None

2.2. Non-Government Documents

ASTM Designation E8	Methods of Tension Testing of Metallic Materials
ASTM Designation E29	Recommended Practices for Designating Significant Places in Specified Limiting Values
ASTM Designation E112	Estimating Average Grain Size of Metals
ASTM Designation E195	Methods for Chemical Analysis of Reactor and Commercial Columbium
GE Specification P10DYA11	Qualification of Vacuum Furnaces for Annealing of Cb-1Zr and T-111 (Ta-8W-2Hf) Alloys
AMS 2635	Radiographic Inspection
GE Specification P3CYA17	Fluorescent Penetrant Inspection
GE Specification P3AYA15	Ultrasonic Method of Inspection
GE Specification P4AYA20	Chemical Cleaning of Columbium, Tantalum, and Their Alloys
GE Specification P4AYA21	Chemical Removal of Nonrefractory Metallic Contamination from Columbium, Tantalum, and Their Alloys

3. REQUIREMENTS

3.1. Vendor Quotations. Vendor quotations will state that the material quoted will be produced in accordance with this specification.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been vacuum melted by the electron beam (preferred but not mandatory) followed by double consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing, and drawing equipment normally found in primary ferrous and nonferrous plants. When canning is used as a means of protection, mild steel is the recommended material to temperatures up to 2300°F, and above 2300°F molybdenum canning is recommended.

3.3. Processing. The starting stock size, processing temperature, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6. and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%, and the amount of final reduction for each mill product (i.e., the reduction imparted between the final in-process recrystallization anneal and the final heat treatment) shall exceed 30%. After the final reduction, the final product shall be given a final vacuum anneal for one hour at a temperature of 3000°F ± 25°F. The amount of total reduction and final reduction shall be reported for each mill product in the certificate of compliance.

3.4. Condition.

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross sectional area to the grain size range specified in paragraph 3.6.

CAUTIONS

Avoid contact between T-111 alloy and copper, nickel, or their alloys except for the copper crucibles used for melting.

Cutting with a cutoff wheel should be avoided if possible. If this operation is required, a silicon carbide wheel (Allison C120-K-RA) must be used. Alumina cutoff wheels should never be used.

3.4.2. Cleaning Prior to Heat Treatment. All material should be cleaned per GE Specification P4AYA21 followed by cleaning per GE Specification P4AYA20 prior to in-process or final heat treatment. Also, all material undergoing a straightening operation following final annealing should be cleaned per GE Specification P4AYA21.

3.4.3. Qualification of Furnaces. Any vacuum furnace to be used for either in-process or final annealing of T-111 must be qualified as explained in GE Specification P10DYA11 to minimize the possibility of contamination.

3.4.4. Heat Treatment. All annealing shall be carried out at pressures of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned per 3.4.2. and protected from furnace parts by a layer of fresh Cb-1Zr (preferred) Cb-1Zr, tantalum or columbium foil 0.002-inch thick or greater. If foil is used for protective wrapping of material to be heat treated, tantalum or Cb-1Zr wire should be used whenever possible for securing of the foil wrap about the material. If spot or tack welding is used for attaching the foil, a molybdenum electrode shall be used and welding contact points shall be at a double thickness of the wrapping material. The ground clamp shall also be a refractory metal or alloy. The use of a copper electrode for spot or tack welding is not permissible. The welding electrode should not come in direct contact with the material or component being prepared for anneal.

When annealing is carried out at pressures greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned Cb-1Zr alloy, tantalum, or columbium retort or wrapped in a minimum of two layers of fresh Cb-1Zr alloy, tantalum, or columbium foil 0.002 inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY	DATE April 21, 1971	NO. B50YA325

3.4.5. Contamination. All items are to be free of surface contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness not greater than [illegible] VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050 inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen, and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.2. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots or billets for conversion to finished products shall be analyzed and must conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot-top-center, top-mid-radius, and topedge, and ingot bottom-center. Duplicate analyses are required for carbon, oxygen, nitrogen, and hydrogen of each location. See paragraph 5.4.1.3. for permissible variations.

3.5.2. Final Production Composition. At the discretion of the purchaser, final products may be analyzed to determine the metallic elements stated in Table I. Failure of the analytical results to conform with the requirements of Table I shall be cause for rejection of all material represented by the analytical sample. If, at the discretion of the purchaser, the final products are not analyzed for metallic elements, the manufacturer's ingot analysis in accordance with paragraph 3.5.1. shall be considered the chemical analysis for the metallic elements of products supplied under this specification. In all cases, duplicate determinations of the elements, carbon, oxygen, nitrogen, and hydrogen shall be performed. The individual values obtained must satisfy the criteria for permissible variations described in paragraph 5.4.1.3. and the average of the values satisfying these criteria must not exceed the limits listed below.

Final Product Concentration Limits, ppm

Element	For Wall Thicknesses 0.020" or Greater Max.	For Wall Thicknesses Less Than 0.020" Max.
Carbon	50	75
Oxygen	150	300
Nitrogen	75	100
Hydrogen	10	10

3.6. Grain Size. The grain size of the final products shall be measured and must conform to the following limits:

SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY	DATE April 21,1971	NO. B50YA325

TABLE I

CHEMICAL COMPOSITION

T-111 (Ta-8W-2Hf) ALLOY

Element	Minimum Concentration ppm	Maximum Concentration ppm
Carbon	-	40
Oxygen	-	100
Nitrogen	-	50
Hydrogen	-	10
Columbium	-	1000
Zirconium	-	500
Molybdenum	-	500
Chromium	-	200
Cobalt	-	50
Iron	-	50
Copper	-	50
Nickel	-	50
Vanadium	-	20
Silicon	-	20
Manganese	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

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Product Wall Thickness, Inches	Minimum Allowable ASTM Grain Size No.	Allowable Spread in ASTM Grain Size Nos. in Any One Item	Minimum Percent Recrystallization
Less than 0.010	7	2	100
> 0.010 to 0.065	6	2	100
> 0.065 to 0.125	5	2	100
> 0.125 to 0.250	4	2	95
> 0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be tested in tension at room temperature (65° - 85°F) and must meet the following property limits:

Ultimate Tensile Strength, ksi		0.2% Yield Strength, ksi		Elongation, %(1)
Minimum	Maximum	Minimum	Maximum	Minimum
80	110	65	100	20

(1) % Elongation in 4D for threaded or buttonhead test specimens; in 1 inch for flat specimens.

3.7.2. Stress-to-Rupture Tests.

3.7.2.1. Test Requirements. Representative samples of the material in final form shall be stress-rupture tested under the following conditions and must have the minimum stress-rupture life shown. For the test results to be valid, the samples must satisfy the environmental contamination criteria of paragraph 3.7.2.2.

Test Temp., °F	Stress, ksi	Minimum Life Hours
2400	19	20

3.7.2.2. Environmental Contamination. Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits.

	Maximum Allowable Increase in Concentration of Impurities, ppm
Carbon	20
Oxygen	35
Nitrogen	20
Hydrogen	1

The maximum allowable increase in the concentration of a given interstitial element shall be the difference between the average concentration of each element in the stress-rupture specimen after testing and the average concentration of that element in the final product from which the stress-rupture sample was taken. The average concentration shall be based on the average of duplicate analytical results. Permissible variations in analytical results are described in paragraph 5.4.1.3.

3.7.3. Hydrostatic Test. Each tube, 1/8-inch or larger in outside diameter with a wall thickness of 0.015-inch or less, shall be tested to a hydrostatic pressure sufficient to product a hoop stress of 50,000 psi. The test pressure, not to exceed 10,000 psi, shall be determined by the equation (P = 2 St/D), where:

P = hydrostatic test pressure in pounds per square inch;

S = 50,000 psi;

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

The test pressure shall be held for a minimum of 5 seconds. After testing, the tubing shall show no bulges, leaks, pinholes, cracks, or other defects.

3.7.4. Flare Test. A section of the heat treated tube shall be flare tested without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 20 percent.

3.8. Tolerances

3.8.1. Diameter and Wall Thickness. The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 8).

3.8.2. Length. When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8-inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4-inch will be permitted, unless otherwise specified.

3.8.3. Straightness. The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600; unless otherwise agreed upon.

4. MAXIMUM ALLOWABLE DISCONTINUITIES

4.1. General. Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. Porosity and Inclusions. Porosity or inclusions with dimensions greater than 3% of the wall thickness or 0.005-inch, whichever is smaller, shall be unacceptable with a minimum rejectable indication of 0.001-inch. Porosity or inclusions with dimensions in the range of 1% to 3% of wall thickness must be a minimum of 0.5-inch apart. Porosity or inclusions with dimensions less than 1% of the wall thickness must be a minimum of 0.12-inch apart.

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TABLE II

PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

Nominal OD, Inches	OD, Inch	ID, Inch	Wall Thickness, %
0.187 to 0.625	± 0.004	± 0.004	± 10
> 0.625 to 1.000	± 0.005	± 0.005	± 10
> 1.000 to 2.000	± 0.0075	± 0.0075	± 10
> 2.000 to 3.000	± 0.010	± 0.010	± 10
> 3.000 to 4.000	± 0.0125	± 0.0125	± 10

NOTES:

(1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.

(2) For tolerances applicable for very small tubes (less than 0.187-inch-diameter) or very thin-wall tubes (less than 0.010-inch-thick), the producer shall be consulted.

(3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3 inch or greater thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.

(4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

4.3. Surface Rework. All surface pores, gouges, cracks and other defects deeper than 0.003-inch or 3% of the wall thickness, whichever is smaller, are unacceptable. Defects may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II (refer to page 8). Grinding should be done with either silicon carbide paper or a silicon carbide grinding wheel. Complete removal of surface defects must be assured by a final swab pickle (to remove smeared metal) with T-111 alloy pickling solution (GE Specification P4AYA20) followed by fluorescent penetrant inspection of the ground area. The defect cannot be considered as being removed until no bleed back of penetrant can be detected following grinding and pickling.

5. QUALITY ASSURANCE PROVISIONS

5.1. Vendor Responsibility. The manufacturer shall make all tests and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. Customer Review. General Electric is to receive advanced notification of all non-proprietary melting, elevated temperature (greater than 500°F) processing operations, testing and nondestructive inspection and is to have the privilege of witnessing said operations. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement of purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operation.

5.3. Sample Selection. Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken transverse to the final working direction from plate and sheet and from strip if of sufficient width. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraph 5.4.2. and 5.4.3. shall be mutually agreed upon by the vendor and purchaser prior to placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analyses

5.4.1.1. Test Procedures. Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM Designation E195, "Methods for Chemical Analysis of Reactor and Commerical Columbium." Specifically, the combustion/conductometric method for carbon; the vacuum fusion method for oxygen and hydrogen; the micro-Kjeldahl method for nitrogen or the carrier gas fusion method for nitrogen and oxygen; the emission spectrographic methods for metallic impurities; the X-ray fluorescence method (using a briquette prepared from the fully oxidized sample) for tungsten; and the colorimetric or atomic absorption method for hafnium. The analytical techniques to be used shall be mentioned in vendor quotations.

SEAMLESS TUBING AND PIPE: T-.. (Ta-8W-2Hf) ALLOY	DATE April 21,1971	NO. B50YA325

5.4.1.2. Check Analyses. Check analyses (see paragraph 7.2.) may be performed by the buyer at his discretion and must confirm that the impurities are within specified limits. Referee (see paragraph 5 3.) analyses shall be used to resolve disputes arising from differences in vendor and buyer analyses.

5.4.1.3. Permissible Variations in Analytical Results. Results of analytical determinations for the interstitial elements, oxygen, carbon, hydrogen, or nitrogen, must be within the permissible range indicated below:

Average Concentration of a Given Interstitial Element, ppm	Permissible Variation from the Average of Duplicate Analytical Results
0-10	± 80%
11-40	± 40%
41-100	± 20%
101-150	± 15%
151-1000	± 10%

In the event that the values are not acceptable, an additional duplicate analysis shall be performed. All values obtained (original duplicate plus second duplicate) shall be averaged and the permissible variaions criteria applied. The average of all acceptable values, two minimum, shall be used in determining the average concentration.

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested at pressures of 1 x 10^{-6} torr or less. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required on Final Product

5.5.1. Destructive Evaluation. Representative test specimens from the finished product representing each ingot and each lot of material shall be tested to determine conformity to this specification. The minimum frequency of these tests shall be:

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Interstitial (Carbon, Oxygen, Hydrogen, Nitrogen) Analyses - two per lop per ingot. See paragraph 3.5.2.

Hafnium Analyses - one per lot per ingot. See paragraph 3.5.2.

Tensile Test - two per lc+ per ingot. See paragraph 3.7.1.

Stress-Rupture Test - two per lop per ingot. See paragraph 3.7.2.

Flare Test - two per lot. per ingot See paragraph 3.7.4.

Grain Size - two per lot per ingot (One longitudinal and one transverse) See paragraph 3.6.

Microhardness Traverses - one per lot per ingot. See paragraph 3.4.5.

5.5.2. Nondestructive Evalu-tion. The following nondestructive inspections shall be performed 100% on all final products unless otherwise specified:

Ultrasonic Inspection. See paragraph 5.7.1.2.

Fluorescent Penetrant Inspection. See paragraph 5.7.1.3.

Hydrostatic Proof Test See paragraph 3.7.3.

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. Inspection.

5.7.1. Methods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2. using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, final products shall be 100% inspected ultrasonically in accordance with GE Specification P3AYA15, "Ultrasonic Method of Inspection," using a 3% notch criteria. Ultrasonic indications having any of the following three descriptions are considered rejectable and could be cause for rejection of the entire piece in which the indication appears:

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1. The amplitude of an ultrasonic signal from the material is = 80% of full screen.

2. The amplitude of an ultrasonic signal from the material is 25% to 79% of full screen and the signal response indicates the source of the signal is 1/8-inch long.

3. Two ultrasonic signals having amplitudes 25% which emanate from sources which are within 1/4-inch of each other.

5.7.1.3. Penetrant Inspection. The exterior surface of products shall be 100% penetrant inspected and found free of flaws as specified in paragraphs 4.1 and 4.3 using GE Specification P3CYA17, "Fluorescent Penetrant Inspection," (Magnaflux penetrant ZL-22A shall be used). All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of GE-NSP "Material Test Summary" forms for each lot of material in place of or in addition to standard vendor test certification forms. The "Material Test Summary" forms summarize all test information required by this specification. Copies of the forms will be supplied to the vendor by GE-NSP.

5.8. Rejections. To be handled in accordance with the Rejections Article of the Conditions of Purchase which appear on the reverse side of the Purchase Order Form.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance. In the event that a referee cannot be mutually agreed upon, the General Electric test results will prevail in determin g the acceptability of the material.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carbon shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order, or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carbon consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

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7. **DEFINITIONS**

7.1. Lot. A lot shall include all material of the same size, shape, condition, and finished from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge, and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile, and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

7.2. Check Analysis. A check analysis is an analysis in addition to the certificated analysis. It shall be performed by the purchaser of the metal, his designated agent, or the referee in case of dispute (see paragraph 5.9.). Permissible variations in the analytical results of the check analyses must conform to those described in paragraph 5.4.1.3. These variations allow for a maximum scatter of analytical data from a specific sample for the given element under reproducible analytical conditions.

7.3. Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29, "Recommended Practices for Designated Significant Places in Specified Limiting Values."

Test	Rounded-Off Unit for Observed or Calculated Value
Chemical Composition and Dimensional Tolerances (When Expressed Decimally)	Nearest Unit in the Last Right-Hand Place of Figures of the Specified Limit
Tensile Strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture Life	Nearest 0.1 Hour

GENERAL ELECTRIC Nuclear Systems Programs Cincinnati, Ohio 45215	SPECIFICATION NO. B50YA325-S1
ENGINEERING SPECIFICATION	ISSUE DATE April 21, 1971

TITLE	ORIGINAL CONTRACT
SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY	SECURITY CLASSIFICATION

Prepared by: R.A. Ekvall Date 4/21/71

Approved by: W.R. [illegible] Date 4/23/71

Approved by: ______ Date ______

Issued by: [illegible] 4/23/71 DRAFTING Mail Zone 26 Phone 5229/5514

SUPERSEDES SPECIFICATION NO	DATED	REVISION AUTHORIZATION	TYPE REVISION SYSTEM
B50YA325	1-7-71		CN CID

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1. SCOPE

1.1. Scope. This specification covers T-111 alloy in tube and pipe form intended for high-temperature structural application and alkali metal containment.

2. APPLICABLE DOCUMENTS

The latest revisions of the following documents apply.

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM Designation E8	Methods of Tension Testing of Metallic Materials
ASTM Designation E29	Recommended Practices for Designating Significant Places in Specified Limiting Values
ASTM Designation E112	Estimating Average Grain Size of Metals
ASTM Designation E195	Methods for Chemical Analysis of Reactor and Commercial Columbium
GE Specification P10DYA11	Qualification of Vacuum Furnaces for Annealing of Cb-1Zr and T-111 (Ta-8W-2Hf) Alloys
AMS 2635	Radiographic Inspection
GE Specification P3CYA17	Fluorescent Penetrant Inspection
GE Specification P3AYA15	Ultrasonic Method of Inspection
GE Specification P4AYA20	Chemical Cleaning of Columbium, Tantalum, and Their Alloys
GE Specification P4AYA21	Chemical Removal of Nonrefractory Metallic Contamination from Columbium, Tantalum, and Their Alloys

3. REQUIREMENTS

3.1. Vendor Quotations. Vendor quotations will state that the material quoted will be produced in accordance with this specification.

3.2. Manufacture. Material covered by this specification shall be made from ingots which have been vacuum melted by the electron beam (preferred but not mandatory) followed by double consumable electrode arc melting processes. Breakdown operations shall be performed with conventional extrusion, forging, tube reducing, and drawing equipment normally found in primary ferrous and nonferrous plants. When canning is used as a means of protection, mild steel is the recommended material to temperatures up to 2300°F, and above 2300°F molybdenum canning is recommended.

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3.3. Processing. The starting stock size, processing temperature, percentages of reduction, in-process annealing temperatures and times shall be selected by the vendor to achieve the grain size range specified in paragraph 3.6. and mechanical properties specified in paragraph 3.7. The total amount of reduction from the turned ingot to the final product shall exceed 75%, and the amount of final reduction for each mill product (i.e., the reduction imparted between the final in-process recrystallization anneal and the final heat treatment) shall exceed 30%. After the final reduction, the final product shall be given a final vacuum anneal for one hour at a temperature of 3000°F ± 25°F. The amount of total reduction and final reduction shall be reported for each mill product in the certificate of compliance.

3.4. Condition.

3.4.1. General. The finished product will be supplied in the recrystallized condition throughout the cross sectional area to the grain size range specified in paragraph 3.6.

CAUTIONS

Avoid contact between T-111 alloy and copper, nickel, or their alloys except for the copper crucibles used for melting.

Cutting with a cutoff wheel should be avoided if possible. If this operation is required, a silicon carbide wheel (Allison C120-K-RA) must be used. Alumina cutoff wheels should never be used.

3.4.2. Cleaning Prior to Heat Treatment. All material should be cleaned per GE Specification P4AYA21 followed by cleaning per GE Specification P4AYA20 prior to in-process or final heat treatment. Also, all material undergoing a straightening operation following final annealing should be cleaned per GE Specification P4AYA21.

3.4.3. Qualification of Furnaces. Any vacuum furnace to be used for either in-process or final annealing of T-111 must be qualified as explained in GE Specification P10DYA11 to minimize the possibility of contamination.

3.4.4. Heat Treatment. All annealing shall be carried out at pressures of less than 1×10^{-5} torr. All mill products to be annealed shall be thoroughly degreased, chemically cleaned per 3.4.2. and protected from furnace parts by a layer of fresh Cb-1Zr (preferred) Cb-1Zr, tantalum or columbium foil 0.002-inch thick or greater. If foil is used for protective wrapping of material to be heat treated, tantalum or Cb-1Zr wire should be used whenever possible for securing of the foil wrap about the material. If spot or tack welding is used for attaching the foil, a molybdenum electrode shall be used and welding contact points shall be at a double thickness of the wrapping material. The ground clamp shall also be a refractory metal or alloy. The use of a copper electrode for spot or tack welding is not permissible. The welding electrode should not come in direct contact with the material or component being prepared for anneal.

When annealing is carried out at pressures greater than 1×10^{-5} torr, with the prior approval of the purchaser, all mill products shall be enclosed in a chemically cleaned Cb-1Zr alloy, tantalum, or columbium retort or wrapped in a minimum of two layers of fresh Cb-1Zr alloy, tantalum, or columbium foil 0.002 inch thick or greater. The conditions of final annealing shall be reported in the certificate of compliance.

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3.4.5. Contamination. All items are to be free of surface contamination or internal oxidation. After final heat treatment, the material shall be examined metallographically for evidence of possible contamination caused by unsatisfactory heat treating atmospheres or processing conditions. A microhardness traverse shall show a hardness not greater than 30 VHN from the center to the surface of a cross sectional sample of the final product. At the discretion of the purchaser, samples taken to include at least one surface of the final product, and not exceeding 0.050 inch thick, may be chemically analyzed by the purchaser for oxygen, nitrogen, hydrogen, and carbon. The analyses shall not exceed the limits set forth in paragraph 3.5.2. Any indication of contamination shall be cause for rejection of all material represented by that sample. The material shall be acceptable if the contaminated layer is completely eliminated before shipment by a machining operation within the specified dimensions and tolerances.

3.5. Chemical Composition

3.5.1. Ingot/Billet Composition. The chemical composition of ingots or billets for conversion to finished products shall be analyzed and must conform to Table I (page 5). A minimum of four analyses shall be obtained as follows: ingot-top-center, top-mid-radius, and topedge, and ingot bottom-center. Duplicate analyses are required for carbon, oxygen, nitrogen, and hydrogen of each location. See paragraph 5.4.1.3. for permissible variations.

3.5.2. Final Production Composition. At the discretion of the purchaser, final products may be analyzed to determine the metallic elements stated in Table I. Failure of the analytical results to conform with the requirements of Table I shall be cause for rejection of all material represented by the analytical sample. If, at the discretion of the purchaser, the final products are not analyzed for metallic elements, the manufacturer's ingot analysis in accordance with paragraph 3.5.1. shall be considered the chemical analysis for the metallic elements of products supplied under this specification. In all cases, duplicate determinations of the elements, carbon, oxygen, nitrogen, and hydrogen shall be performed. The individual values obtained must satisfy the criteria for permissible variations described in paragraph 5.4.1.3. and the average of the values satisfying these criteria must not exceed the limits listed below.

Final Product Concentration Limits, ppm

	For Wall Thicknesses 0.020" or Greater	For Wall Thicknesses Less Than 0.020"
Element	Max.	Max.
Carbon	50	75
Oxygen	150	200
Nitrogen	75	100
Hydrogen	10	10

3.6. Grain Size. The grain size of the final products shall be measured and must conform to the following limits:

TABLE I

CHEMICAL COMPOSITION

T-111 (Ta-8W-2Hf) ALLOY

Element	Minimum Concentration ppm	Maximum Concentration ppm
Carbon	-	40
Oxygen	-	100
Nitrogen	-	50
Hydrogen	-	10
Columbium	-	1000
Zirconium	-	500
Molybdenum	-	500
Chromium	-	200
Cobalt	-	50
Iron	-	50
Copper	-	50
Nickel	-	50
Vanadium	-	20
Silicon	-	20
Manganese	-	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	-

Product Wall Thickness, Inches	Minimum Allowable ASTM Grain Size No.	Allowable Spread in ASTM Grain Size Nos. in Any One Item	Minimum Percent Recrystallization
Less than 0.010	7	2	100
> 0.010 to 0.065	6	2	100
> 0.065 to 0.125	5	2	100
> 0.125 to 0.250	4	2	95
> 0.250 to 0.500	3	3	90

3.7. Mechanical Properties. The final product shall satisfy the following mechanical property requirements:

3.7.1. Room Temperature Tensile Properties. Representative samples of the material in final form shall be tested in tension at room temperature (65° - 85°F) and must meet the following property limits:

Ultimate Tensile Strength, ksi		0.2% Yield Strength, ksi		Elongation, %(1)
Minimum	Maximum	Minimum	Maximum	Minimum
80	110	65	100	20

(1) % Elongation in 4D for threaded or buttonhead test specimens; in 1 inch for flat specimens.

3.7.2. Stress-to-Rupture Tests.

3.7.2.1. Test Requirements. Representative samples of the material in final form shall be stress-rupture tested under the following conditions and must have the minimum stress-rupture life shown. For the test results to be valid, the samples must satisfy the environmental contamination criteria of paragraph 3.7.2.2.

Test Temp., °F	Stress, ksi	Minimum Life Hours
2400	19	20

3.7.2.2. Environmental Contamination. Chemical analysis of stress-rupture specimens after test shall demonstrate that the degree of environmental contamination did not exceed the following limits.

	Maximum Allowable Increase in Concentration of Impurities, ppm
Carbon	20
Oxygen	35
Nitrogen	20
Hydrogen	4

The maximum allowable increase in the concentration of a given interstitial element shall be the difference between the average concentration of each element in the stress-rupture specimen after testing and the average concentration of that element in the final product from which the stress-rupture sample was taken. The average concentration shall be based on the average of duplicate analytical results. Permissible variations in analytical results are described in paragraph 5.4.1.3.

3.7.3. <u>Hydrostatic Test.</u> Each tube, 1/8-inch or larger in outside diameter with a wall thickness of 0.015-inch or less, shall be tested to a hydrostatic pressure sufficient to product a hoop stress of 50,000 psi. The test pressure, not to exceed 10,000 psi, shall be determined by the equation ($P = 2\ St/D$), where:

P = hydrostatic test pressure in pounds per square inch;

S = 50,000 psi;

t = average wall thickness of the tube in inches;

D = outside diameter of the tube in inches.

The test pressure shall be held for a minimum of 5 seconds. After testing, the tubing shall show no bulges, leaks, pinholes, cracks, or other defects.

3.7.4. <u>Flare Test.</u> A section of the heat treated tube shall be flare tested without cracking. The flare shall be made with a tool having a 60-degree included angle until the specified outside diameter has been increased by 20 percent.

3.8. <u>Tolerances</u>

3.8.1. <u>Diameter and Wall Thickness.</u> The permissible variations in diameter and wall thickness of tube shall not exceed those prescribed in Table II (refer to page 8).

3.8.2. <u>Length.</u> When tube is ordered cut-to-length, the usable length shall not be less than that specified, but a variation of plus 1/8-inch will be permitted in lengths up to 6 feet. In lengths over 6 feet, a variation of plus 1/4-inch will be permitted, unless otherwise specified.

3.8.3. <u>Straightness.</u> The tube shall be free of bends or kinks. For lengths up to 10 feet, the maximum bow shall not exceed one part in 1200; for lengths greater than 10 feet, the maximum bow shall not exceed one part in 600; unless otherwise agreed upon.

4. <u>MAXIMUM ALLOWABLE DISCONTINUITIES</u>

4.1. <u>General.</u> Cracks, laps, seams, fins, and tears shall be unacceptable. The surface shall also be free from oxide or scale of any nature, grease, oil, residual lubricants, or other extraneous material.

4.2. <u>Porosity and Inclusions.</u> Porosity or inclusions with dimensions greater than 3% of the wall thickness or 0.005-inch, whichever is smaller, shall be unacceptable with a minimum rejectable indication of 0.001-inch. Porosity or inclusions with dimensions in the range of 1% to 3% of wall thickness must be a minimum of 0.5-inch apart. Porosity or inclusions with dimensions less than 1% of the wall thickness must be a minimum of 0.12-inch apart.

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TABLE II

PERMISSIBLE VARIATIONS IN TUBE DIMENSIONS

Nominal OD, Inches	OD, Inch	ID, Inch	Wall Thickness, %
0.187 to 0.625	± 0 004	± 0.004	± 10
> 0.625 to 1.000	± 0.005	± 0.005	± 10
> 1.000 to 2.000	± 0.0075	± 0.0075	± 10
> 2.000 to 3.000	± 0.010	± 0.010	± 10
> 3.000 to 4.000	± 0.0125	± 0.0125	± 10

NOTES:

(1) Tolerances are applicable to only the two dimensions specified on the purchase order, e.g., outside diameter and wall; inside diameter and wall; outside diameter and inside diameter.

(2) For tolerances applicable for very small tubes (less than 0.187-inch-diameter) or very thin-wall tubes (less than 0.010-inch-thick), the producer shall be consulted.

(3) For tubes having an inside diameter less than 60% of the outside diameter or a wall 3/4 inch or over thick, which cannot be successfully drawn over a mandrel, the inside diameter may vary by an amount equal to plus or minus 10% of the wall thickness. The wall thickness of these tubes may vary plus or minus 12.5% from that specified.

(4) Ovality measured at any cross section: For tubes with nominal wall thickness less than 3% of the nominal outside diameter, the ovality tolerances are double the tolerances in column 2 or 3. For ovality tolerances for tubes with wall thickness less than 2% nominal outside diameter, the producer shall be consulted.

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4.3. **Surface Rework.** All surface pores, gouges, cracks and other defects deeper than 0.005-inch or 3% of the wall thickness, whichever is smaller, are unacceptable. Defects may be removed by grinding provided the wall thickness is not decreased below that permitted in Table II (refer to page 8). Grinding should be done with either silicon carbide paper or a silicon carbide grinding wheel. Complete removal of surface defects must be assured by a final swab pickle (to remove smeared metal) with T-111 alloy pickling solution (GE Specification P4AYA20) followed by fluorescent penetrant inspection of the ground area. The defect cannot be considered as being removed until no bleed back of penetrant can be detected following grinding and pickling.

5. QUALITY ASSURANCE PROVISIONS

5.1. **Vendor Responsibility.** The manufacturer shall make all tests and inspections of the material covered by this specification before shipment unless otherwise specified. All test and inspection results shall be furnished to the purchaser.

5.2. **Customer Review.** General Electric is to receive advanced notification of all non-proprietary melting, elevated temperature (greater than 500°F) processing operations, testing and nondestructive inspection and is to have the privilege of witnessing said operations. The manufacturer shall give the purchaser ample notice of the time and place of designated tests. If the purchaser's representative is not present at this time and a new date is not set, the requirement of purchaser's inspection at the place of testing is waived. When the purchaser's representative is present at the appointed time and place, the manufacturer shall afford him, without charge, all reasonable facilities to assure that the material is being furnished in accordance with this specification. This inspection shall not interfere unnecessarily with production operation.

5.3. **Sample Selection.** Care shall be exercised to insure that the sample selected for testing is representative of the material and uncontaminated by the sampling procedure. Samples for the determination of mechanical properties shall be selected so as to consume a minimum amount of material, i.e., specimens may be taken transverse to the final working direction from plate and sheet and from strip if of sufficient width. If there is any question about the sampling technique or the analysis, the methods for sampling and analysis shall be those agreed to by the buyer and seller. The specimen configuration selected for the performance of the testing required in paragraph 5.4.2. and 5.4.3. shall be mutually agreed upon by the vendor and purchaser prior to placement of a purchase order. The location of all test samples shall be reported in the certificate of compliance.

5.4. Test Methods

5.4.1. Chemical Analyses

5.4.1.1. **Test Procedures.** Chemical analyses shall be conducted by mutually acceptable procedures, such as the vacuum fusion methods for gases, the combustion method for carbon, and spectrochemical methods for metallic elements. Disputes shall be settled by accepted referee methods, such as the ASTM Designation E195, "Methods for Chemical Analysis of Reactor and Commerical Columbium." Specifically, the combustion/conductometric method for carbon, the vacuum fusion method for oxygen and hydrogen; the micro Kjeldahl method for nitrogen or the carrier gas fusion method for nitrogen and oxygen; the emission spectrographic methods for metallic impurities; the X-ray fluorescence method (using a briquette prepared from the fully oxidized sample) for tungsten; and the colorimetric or atomic absorption method for hafnium. The analytical techniques to be used shall be mentioned in vendor quotations.

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5.4.1.2. Check Analyses. Check analyses (see paragraph 7.2.) may be performed by the buyer at his discretion and must confirm that the impurities are within specified limits. Referee (see paragraph 5.9.) analyses shall be used to resolve disputes arising from differences in vendor and buyer analyses.

5.4.1.3. Permissible Variations in Analytical Results. Results of analytical determinations for the interstitial elements, oxygen, carbon, hydrogen, or nitrogen, must be within the permissible range indicated below:

Average Concentration of a Given Interstitial Element, ppm	Permissible Variation from the Average of Duplicate Analytical Results
0-10	± 80%
11-40	± 40%
41-100	± 20%
101-150	± 15%
151-1000	± 10%

In the event that the values are not acceptable, an additional duplicate analysis shall be performed. All values obtained (original duplicate plus second duplicate) shall be averaged and the permissible variaions criteria applied. The average of all acceptable values, two minimum, shall be used in determining the average concentration.

5.4.2. Tensile Test. The tension test shall be conducted in accordance with ASTM Designation E8, "Methods of Tension Testing of Metallic Materials." Yield strength shall be determined by the offset (0.2%) method. The tensile properties shall be determined using a strain rate of 0.005 inch per inch per minute up to 0.6% offset and then 0.050 inch, plus or minus 0.020 inch, per inch per minute to fracture.

5.4.3. Stress-Rupture Test. Stress-rupture properties of specimens shall be determined by mutually acceptable testing techniques. Suggested testing techniques for determining stress-rupture properties are:

Specimens shall be tested at pressures of 1×10^{-6} torr or less. The vacuum system shall incorporate an optically tight liquid nitrogen cold trap or a getter-ion pump.

Specimens shall be held for a half hour at the test temperature before application of load.

Test temperature shall be maintained at plus or minus 10°F during the test.

5.4.4. Grain Size. Grain size determinations shall be made according to ASTM Specification E112, "Estimating the Average Grain Size of Metals."

5.5. Number of Tests Required on Final Product

5.5.1. Destructive Evaluation. Representative test specimens from the finished product representing each ingot and each lot of material shall be tested to determine conformity to this specification. The minimum frequency of these tests shall be:

Interstitial (Carbon, Oxygen, Hydrogen, Nitrogen) Analyses - two per lop per ingot. See paragraph 3.5.2.

Hafnium Analyses - one per lot per ingot. See paragraph 3.5.2.

Tensile Test - two per lot per ingot. See paragraph 3.7.1.

Stress-Rupture Test - two per lop per ingot. See paragraph 3.7.2.

Flare Test - two per lot, per ingot See paragraph 3.7.4.

Grain Size - two per lot per ingot (One longitudinal and one transverse) See paragrapn 3.6.

Microhardness Traverses - one per lot per ingot. See paragraph 3.4.5.

5.5.2. Nondestructive Evaluation. The following nondestructive inspections shall be performed 100% on all final products unless otherwise specified:

Ultrasonic Inspection. See paragraph 5.7.1.2.

Fluorescent Penetrant Inspection. See paragraph 5.7.1.3.

Hydrostatic Proof Test See paragraph 3.7.3.

5.6. Retest and Rework

5.6.1. Surface Contamination. Any sample or specimen exhibiting obvious surface contamination or improper preparation which disqualifies it as a truly representative sample shall be replaced with a new sample.

5.6.2. Rework. If inspection and test results of a lot do not conform to the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if all test results, after reworking, conform to this specification.

5.7. ction.

5.7.1 nods of Inspection

5.7.1.1. Radiographic. When specified, the product shall be radiographed and found free of porosity and inclusions as specified in paragraph 4.2. using the techniques described in AMS 2635, "Radiographic Inspection." The radiographs and product shall be identified so that the exact position of each radiograph can be correlated with the specific area on a particular product.

5.7.1.2. Ultrasonic Inspection. Unless otherwise agreed to by the purchaser and the vendor, final products shall be 100% inspected ultrasonically in accordance with GE Specification P3AYA15, "Ultrasonic Method of Inspection," using a 3% notch criteria. Ultrasonic indications having any of the following three descriptions are considered rejectable and could be cause for rejection of the entire piece in which the indication appears:

1. The amplitude of an ultrasonic signal from the material is - 80% of full screen.

2. The amplitude of an ultrasonic signal from the material is 25% to 79% of full screen and the signal response indicates the source of the signal is 1/8-inch long.

3. Two ultrasonic signals having amplitudes 25% which emanate from sources which are within 1/4-inch of each other.

5.7.1.3. Penetrant Inspection. The exterior surface of products shall be 100% penetrant inspected and found free of flaws as specified in paragraphs 4.1 and 4.3 using GE Specification P3CYA17, "Fluorescent Penetrant Inspection," (Magnaflux penetrant ZL-22A shall be used). All parts thus inspected shall be marked with ink stamps as described in the specification; impression stampings or etching shall be unacceptable.

5.7.1.4. Reports. The manufacturer shall supply at least three copies of GE-NSP "Material Test Summary" forms for each lot of material in place of or in addition to standard vendor test certification forms. The "Material Test Summary" forms summarize all test information required by this specification. Copies of the forms will be supplied to the vendor by GE-NSP.

5.8. Rejections. To be handled in accordance with the Rejections Article of the Conditions of Purchase which appear on the reverse side of the Purchase Order Form.

5.9. Referee. If the manufacturer and the purchaser disagree concerning the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee's test shall be used to determine conformance. In the event that a referee cannot be mutually agreed upon, the General Electric test results will prevail in determining the acceptability of the material.

6. PREPARATION FOR DELIVERY

6.1. Identification. Each bundle, box, or carbon shall be legibly and conspicuously marked or tagged with the number of this specification, purchase order, or contract number, type, ingot number, lot number, nominal size, and the gross, net, and tare weights. When each bundle, box or carbon consists of components from more than one ingot number or lot number, each component shall be identified individually.

6.2. Packing. The ends of each pipe or tube shall be sealed with suitable plastic caps and each individual item shall be wrapped in heavy gauge polyethylene or similar material and packed in a manner assuring safe delivery when properly transported by a common carrier.

SEAMLESS TUBING AND PIPE: T-111 (Ta-8W-2Hf) ALLOY	DATE April 21,1971	NO B50YA325

7. DEFINITIONS

7.1. Lot. A lot shall include all material of the same size, shape, condition, and finished from one heat of material and which has received the same processing, has been annealed in the same vacuum annealing charge, and has been processed simultaneously in all operations in which temperatures may reach 500°F or above. When process temperatures and environments are closely controlled or when closely adjacent sizes receive similar processing, lots may be combined for chemical, tensile, and stress-rupture tests only, provided prior written approval has been obtained from the General Electric Company.

Check Analysis. A check analysis is an analysis in addition to the certificated analysis. It shall be performed by the purchaser of the metal, his designated agent, or the referee in case of dispute (see paragraph 5.9.). Permissible variations in the analytical results of the check analyses must conform to those described in paragraph 5.4.1.3. These variations allow for a maximum scatter of analytical data from a specific sample for the given element under reproducible analytical conditions.

Significance of Numerical Limits. For determining compliance with the specified limits for requirements of the properties listed below, an observed value or a calculated value shall be rounded off using the rounding-off method in ASTM Designation E29, "Recommended Practices for Designated Significant Places in Specified Limiting Values."

Test	Rounded-Off Unit for Observed or Calculated Value
Chemical Composition and Dimensional Tolerances (When Expressed Decimally)	Nearest Unit in the Last Right-Hand Place of Figures of the Specified Limit
Tensile Strength	Nearest 100 psi
Elongation	Nearest 1%
Rupture Life	Nearest 0.1 Hour

APPENDIX D

PROCESSING OF T-111 ALLOY

FANSTEEL METALLURGICAL CORPORATION

The major portion of the T-111 alloy mill products was produced by Fansteel Metallurgical Corporation. In the production of T-111 alloy ingots, tantalum and tungsten powders are blended in one large charge at Fansteel's Muskogee, Oklahoma, facilities. Subsequently, the powders are hydrostatically compacted and sintered into rectangular bars. Consolidation of the T-111 alloy is accomplished first by double EB-melting the sintered Ta-8W bars into 5-inch (12.7-cm) ingots. The hafnium addition to the alloy is accomplished by GTA-welding reactor-grade hafnium strips to the full length of the electrode produced from the 5-inch (12.7-cm)-diameter EB-melted ingots and single vacuum-arc-melting to produce either a 7 1/2-inch (19.1-cm)- or a 9 1/2-inch (24.1-cm)-diameter T-111 alloy ingot. Six arc-cast ingots were required to fulfill the program requirements. Heat numbers and ingot sizes are:

Heat No.	Size	Melting Source
111D1632	9 1/2 in. (24.1 cm)	Braeburn Alloy Steel
111D1633	9 1/2 in. (24.1 cm)	Braeburn Alloy Steel
111D1670	9 1/2 in. (24.1 cm)	Univ. Cyclops Steel
111D1102	9 1/2 in. (24.1 cm)	Univ. Cyclops Steel
111D1765	7 1/2 in. (19.1 cm)	Fansteel
111D1829	7 1/2 in. (19.1 cm)	Fansteel

After machining, the ingots were shipped to Canton Drop Forging Company, Canton, Ohio, canned in Type 304 SS seamless pipe, and extruded. The extrusion parameters are presented in Table D-I.

Heats 111D1632/111D1633

After extrusion, the cans were removed and the billets were conditioned for forging at Anderson-Schumaker, Chicago, Illinois. Forging was to be performed at 2200°-2300°F (1204°-1260°C) on open-face dies using a 6000-pound (2720-kg) hammer; however, inspection of the extrusions revealed extensive cracking along the entire length of both extrusions. A transverse slice of one of the extrusions was forwarded to General Electric for examina-

TABLE D-I

EXTRUSION PARAMETERS FOR T-111 ALLOY INGOTS

Parameter \ Heat No.	111D1632/111D1633	111D1670*	111D1102	111D1765	111D1829
Machined Ingot Dia., In.	8.44	8.44	8.44	6.72	6.72
Ingot Nose Geometry	90° angle	90° angle	90° angle	90° angle	90° angle
Can Material/Size	9-1/16-inch OD x 1/4-inch thick wall Type 304SS seamless pipe with 2-inch thick Type 304SS nose block and 1/2-inch thick Type 304SS back-up block contained within the can with argon purge tubes attached	9-1/16-inch OD x 1/4-inch thick wall Type 304SS seamless pipe with a 2-inch Thick Type 304SS nose block and a 1/2-inch thick Type 304SS back-up block contained within the can	9-1/16-inch OD x 1/4-inch thick wall Type 304 SS seamless pipe with a 2-inch thick Type 304 SS nose block and a 1/2-inch thick Type 304 SS back-up block contained within the can	7-1/4-inch OD x 1/4-inch thick wall Type 304 SS seamless pipe with a molybdenum foil liner, a 2-inch thick Type 304 SS nose block and a 1/2-inch thick Type 304 SS back-up block contained within the can	7-1/4-inch OD x 1/4-inch thick wall Type 304 SS seamless pipe with a molybdenum foil liner, a 2-inch thick Type 304 SS nose block and 1/2-inch thick Type 304 SS back-up block contained within the can with argon purge tubes attached
Leader Block/Follow-up Block	9-1/16-inch diameter x 6-inch long mild steel	9-1/16-inch diameter x 6-inch long mild steel	9-1/16-inch diameter x 6-inch long mild steel	7-1/16-inch diameter x 6-inch long mild steel	7-1/16-inch diameter x 6-inch long mild steel
Container ID, In.	9-1/4	9-1/4	9-1/4	7.51	7.51
Die Dia., In./Design	5-1/8/conical	5-1/8/conical	5-1/8/conical	4.5/conical	4.5/conical
Die Coating	None	None	None	None	None
Extrusion Ratio	3.2/1	3.2/1	3.2/1	2.7/1	2.7/1
Lubricant	Hot die grease similar to Fiske 604	Hot die grease similar to Fiske 604	Hot die grease similar to Fiske 604	Hot die grease similar to Fiske 604	Hot die grease similar to Fiske 604
Furnace Temperature, °F/Soak Time in Salt Bath, Hours	2200/1	2200/45 min. 2300/75 min.	2300/75 min.	2200/2 hrs.	2200/2 hrs.
Handling Time, Sec.	60				
Average Extrusion Pressure(Hydraulic)psi	2900 (Peak) 2700 (Runout)	2500 (Peak) 2400 (Runout)	Billet A - 2300 (Peak) 2200 (Runout) Billet B - 2100 (Peak) 2000 (Runout)	1800 (Peak) 1500 (Runout)	1800 (Peak) 1600 (Runout)
Maximum Allowable Pressure, psi	Approx. 3850	3850	3850	3850	3850
Cooling Procedure	Air Cooled	Air Cooled	Air Cooled	Air Cooled	Air Cooled

*The ingot was 3 inches too long for the container and the pressure had to be stopped before the extrusion was completed. The stem was withdrawn, a pusher block inserted in the container and subsequently the extrusion was completed (approximately 10-second delay). The tail of the extrusion showed cracks similar to those in the first extrusions.

tion. Macrographs of the transverse section, shown in Figure D-1, revealed three major cracks which were intergranular in nature and at approximately 120^{o} intervals around the periphery of the extrusion.

An investigation was performed to delineate the possible cause of the cracking. A Rockwell hardness survey across a transverse slice from one of the first 5 1/8-inch (13-cm)-diameter T-111 alloy extrusions (No. 111D1632), which had cracked, revealed no significant hardness variation across the billet, and the average hardness was found to be 61 R_a. Subsequently, the transverse slice from the extrusion was sectioned, as shown in Figure D-2, to prepare specimens for metallographic examination and chemical analysis. Samples were obtained from the center and from the edge of the extrusion. Photomicrographs of the extrusion are shown in Figures D-3 and D-4, and no major amounts of precipitate or second phases were observed at any of the locations examined.

Chemical analysis results of the slice are shown in Table D-II. Based on the chemical analyses of the one cross section of the extrusion, the ingot appeared to be reasonably homogeneous. The results of chemical analysis do not indicate any obvious concentrations that might contribute to observed cracking.

T-111 alloy rods measuring 2 1/4 inches (5.7 cm) and 3 3/4 inches (9.5 cm) in diameter were salvaged from these two T-111 alloy ingots. The 2 1/4-inch (5.7-cm)-diameter material was processed into 1/8-inch- to 1 1/2-inch (0.32- to 3.8-cm)-diameter rods and 0.062- and 0.094-inch (0.16- and 0.24-cm)-diameter wire; the 3 3/4-inch (9.5-cm)-diameter material was processed into 0.005- and 0.009-inch (0.013- and 0.023-cm)-thick foil, 0.040-inch (0.10-cm)-thick sheet, and 0.500-inch (1.27-cm)-thick plate. Processing temperatures were less than 800^{o}F (427^{o}C). All of these products except the 1 1/2-inch (3.8-cm)-diameter rod were given a final anneal of 2687^{o}F (1475^{o}C) for one hour; the 1 1/2-inch (3.8-cm)-diameter rod was annealed at 2687^{o}F (1475^{o}C) for two hours.

Heat No. 111D1670

After extrusion, the can was removed and the billet was conditioned for forging. No cracks were found on the surface of the extrusion as determined by dye penetrant inspection techniques.

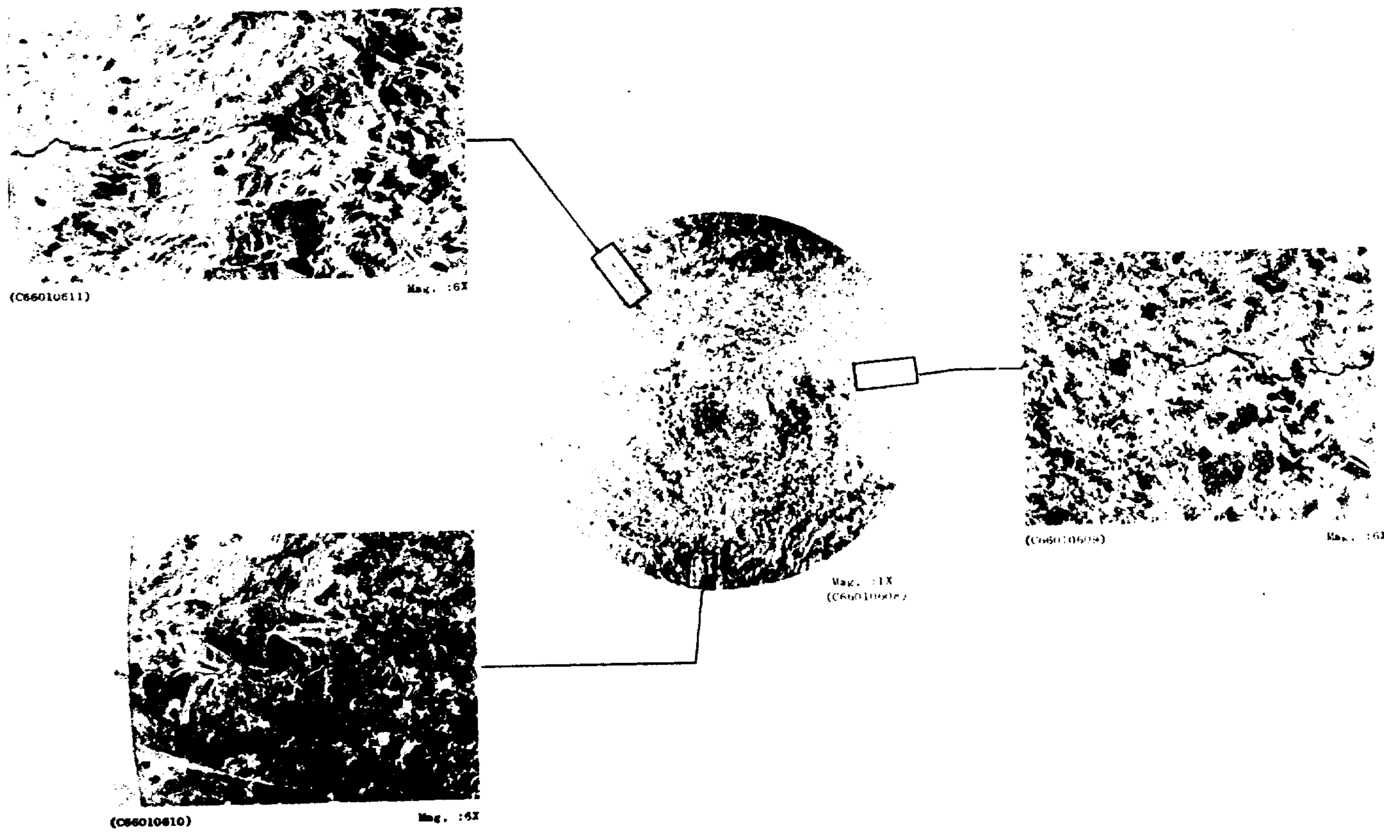


Figure D-1. Macrographs of the T-111 Alloy Extrusion From Heat 111D1632 Showing Intergranular Cracks at Surface at Approximately 120° Intervals. Etchant: 30HD-30HF-15NHO_3

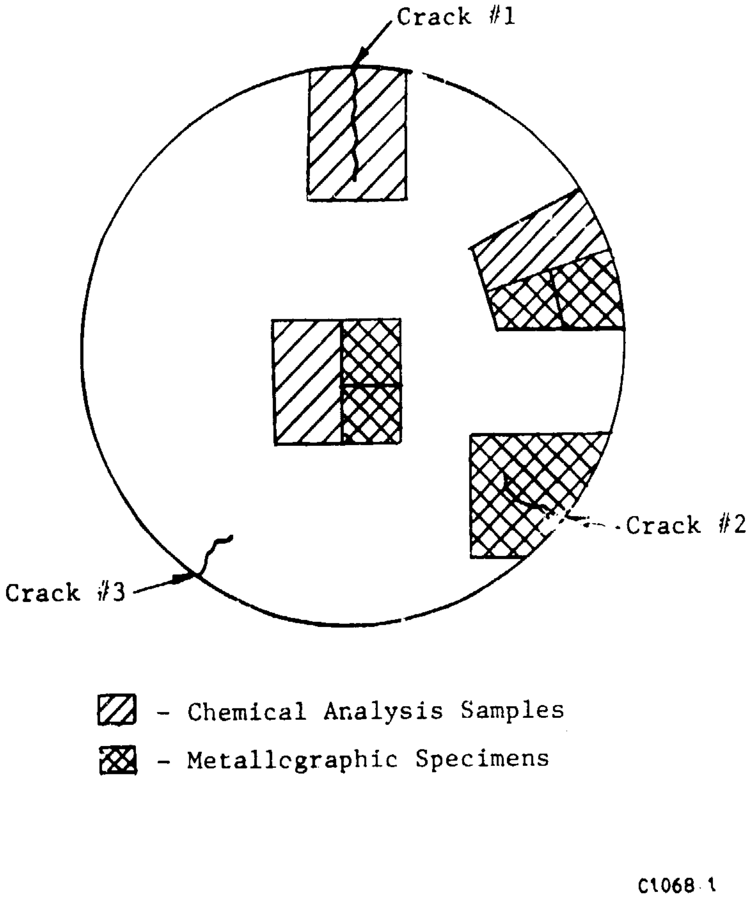


Figure D-2. Sectioning of Transverse Slice from Extrusion of Fansteel Ingot No. 111D1632.

(a) Longitudinal Section

(b) Transverse Section

C1068-2

Figure D-3. The Microstructure of the Center of the Transverse Slice From the T-111 Alloy Extrusion - Fansteel Heat No. 111D1632.

Etchant: 30HCl, 30HF, $15HNO_3$ a) B170511, b) B170411
Mag.: 30X

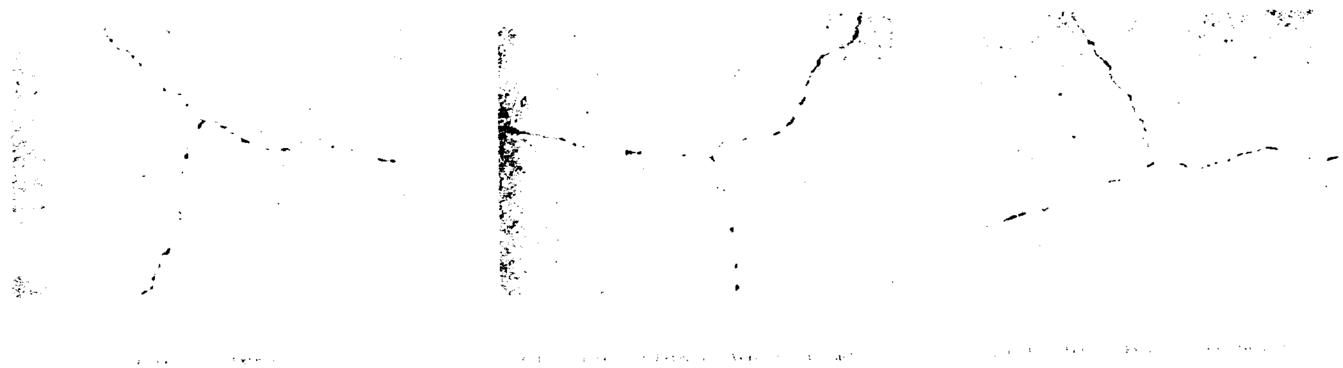


Figure D-4. Microstructures of Transverse Sections From the T-111 Extrusion - Fansteel Heat No. 111D1632. Etchant: Electrolytically Anodized. Mag.: 100X
a) B170413, b) B170213, c) B1706111

TABLE D-II

CHEMICAL ANALYSES OF CRACKED T-111 ALLOY
EXTRUSION - FANSTEEL INGOT NO. 111-D1632

Element	Method of Analyses	Chemical Analyses: Periphery Near Crack	Chemical Analyses: Periphery Away From Crack	Chemical Analyses: Center
W	(1)	7.25%	7.31%	6.97%
Hf	(1)	2.45%	2.32%	2.28%
C	(2)	30 ppm	20 ppm	30 ppm
O	(3)	70 ppm	60 ppm	109 ppm
N	(3)	12 ppm	10 ppm	14 ppm
Ag	(4)	--	--	--
Al	(4)	--	(6)	(6)
B	(4)	--	--	--
Be	(4)	--	--	--
Bi	(4)	--	--	--
Ca	(4)	--	--	--
Cb	(4)	--	--	--
Cd	(4)	--	--	--
Co	(4)	--	--	--
Cr	(4)	--	(7)	(7)
	(5)	10 ppm	--	10 ppm
Cu	(4)	--	--	--
Fe	(4)	--	(6)	(6)
	(5)	64 ppm	--	34 ppm
K	(4)	--	--	--
Mg	(4)	--	(6)	(6)
Mn	(4)	--	(6)	(6)
Mo	(4)	--	--	--
Na	(4)	--	--	--
Ni	(4)	--	(7)	(7)
	(5)	<10 ppm	--	<10 ppm
Pb	(4)	--	--	--
Si	(4)	--	(6)	(6)
Ti	(4)	--	(6)	(6)
V	(4)	--	--	--
Y	(4)	--	--	--
Zn	(4)	--	--	--
Zr	(4)	--	--	--

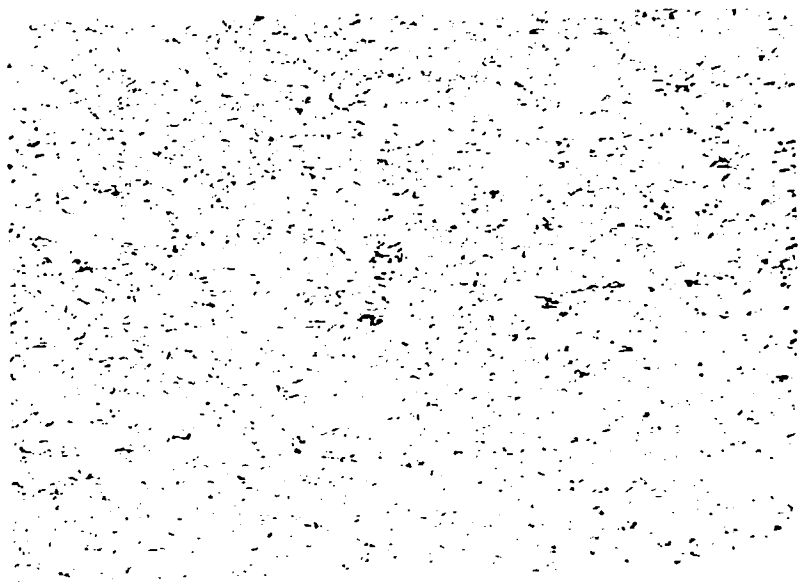
(1) Wet analysis (Ledoux & Co. Inc., Teaneck, N.J.)
(2) Conductometric
(3) Vacuum fusion
(4) Spectrographic (semi-quantitative)
(5) Colormetric (Ledoux & Co. Inc., Teaneck, N.J.)
(6) Trace amount (< 500 ppm)
(7) <50 ppm

NOTE: Semi-quantitative spectrographic analyses revealed no difference in concentration of those elements that were detected between the center and edge of the extrusion.

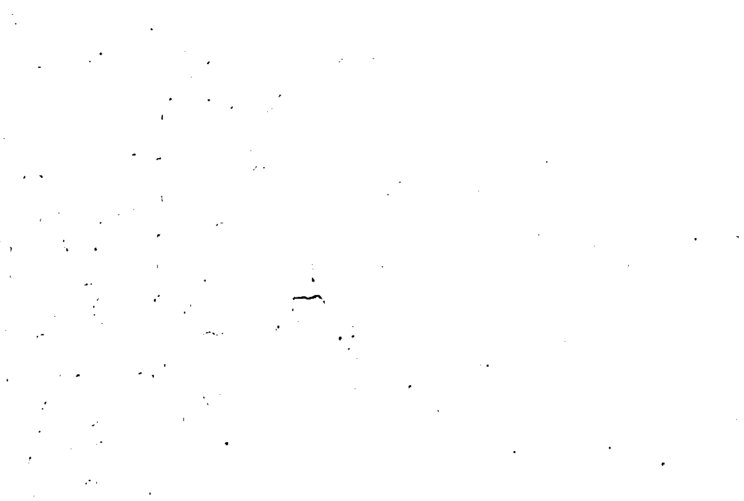
Trial forgings of portions of the extrusion which were conducted at Anderson Schumaker, Chicago, Illinois, and Westinghouse Electric Corporation, Materials Manufacturing Division, Blairsville, Pennsylvania, were unsuccessful due to center cracking of the billets. The trial forging that was attempted at Anderson-Schumaker utilized a 6000-pound (2720-kg) hammer, flat dies, and an initial billet temperature of 2300°F (1260°C). The billet was heated in air without a protective coating. In an attempt to work the center of the billet more extensively, the trial forging conducted at Westinghouse utilized a 1000-ton (9.1×10^5-kg) press. The dies consisted of a flat-top die and a closed-bottom die. The billet was heated to the 2300°F (1260°C) forging temperature in an Inconel retort containing flowing argon gas. In the latter case, the center cracking was confined to the ends. After conditioning to remove any contamination resulting from the previous forging operation, this billet was heated to 1700°F (927°C) in an air furnace and rod-rolled successfully to 2.1-inch (5.3-cm) diameter without reheating. Subsequently, two 4.5-inch (11.4-cm)-diameter billets were rolled successfully in a similar manner to 3 1/8 inches (8.0 cm) in diameter and were utilized for the production of 3/8-inch (0.95-cm)-OD and one-inch (2.5-cm)-OD tubing.

The two 3 1/8-inch (8.0-cm)-diameter rods were machined into 2.93-inch (7.44-cm)-diameter x 9-inch (22.9-cm)-long billets and shipped to Wolverine Tube Division of Calumet-Hecla. The billets were drilled to make tube hollows and were extruded into base tube blanks using procedures developed at Wolverine. The processing procedures are considered proprietary by Wolverine Tube. The base tubes were cleaned, the ID honed, the OD polished, recleaned and inspected. A brief study was performed at General Electric to aid in selecting the annealing temperature to be used on the base tubes prior to tube reducing. Samples from the base tubes were annealed for one hour at 2500°, 2600°, 2700°, 2800°, 2900°, and 3000°F (1371°, 1427°, 1482°, 1538°, 1593°, and 1649°C) in vacuum. The samples were examined by metallographic and microhardness techniques.

As can be seen in Figures D-5 through D-8, recrystallization has occurred in all of the annealed specimens. The microhardness and grain-size data are presented in Table D-III. A one-hour at 2700°F (1482°C) anneal was selected to obtain a desirable combination of hardness and grain size. Subsequently,



a) As Extruded



b) Annealed, 2500°F (1371°C) - 1 Hour

C200"-1

Figure D-5. Transverse Microstructure of T-111 Alloy Extruded Base Tube.

Etchant: 30gmNH_4F-20mlH_2O-50mlHNO_3

Mag.: 100X a) B830114, b) B830211

a) Annealed, 2600°F (1427°C) - 1 Hour

b) Annealed, 2700°F (1482°C) - 1 Hour

C2004-2

Figure D-6. Transverse Microstructure of T-111 Alloy Extruded Base Tube.

Etchant: 30gmNH_4F-20mlH_2O-50mlHNO_3

Mag.: 100X a) B830311, b) B830411

a) Annealed, 2800°F (1538°C) - 1 Hour

b) Annealed, 2900°F (1593°C) - 1 Hour

Figure D-7. Transverse Microstructure of T-111 Alloy Extruded Base Tube.

Etchant: $30gmNH_4F-20mlH_2O-50mlHNO_3$

Mag.: 100X a) B830511, b) B830611

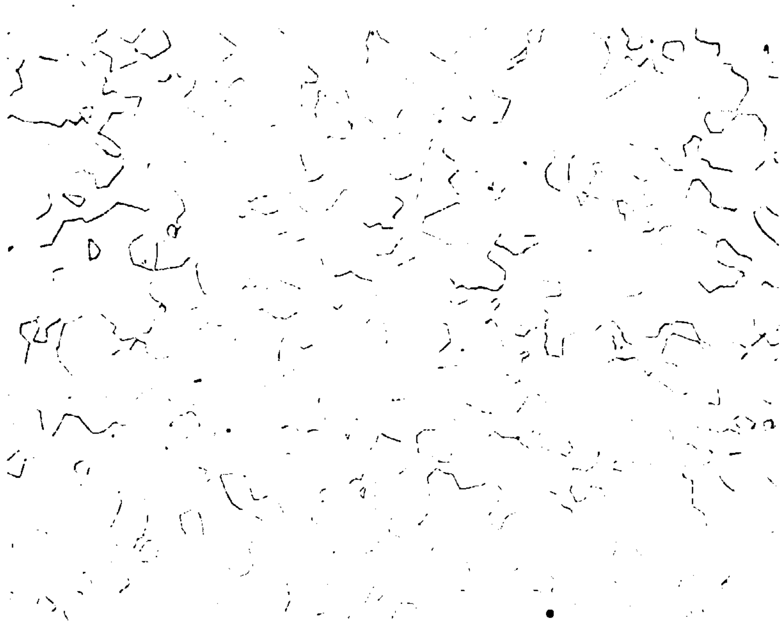


Figure D-8. Transverse Microstructure of T-111 Alloy Extruded Base Tube After Annealing at 3000°F (1649°C) for One Hour in Vacuum.

Etchant: 30gmNH_4F-20mlH_2O-50mlHNO_3

Mag.: 100X (B830711)

TABLE D-111

MICROHARDNESS AND GRAIN SIZE OF UNANNEALED AND ANNEALED[a] T-111 ALLOY HEAT NUMBER 111-D-1670 EXTRUDED BASE TUBE

Annealing Temperature °F	°C	Microhardness[b] DPH	ASTM Grain Size[c]
As Extruded		314	(d)
2500	1316	217	7.5
2600	1427	212	7.5
2700	1482	210	7.5
2800	1538	216	7.0
2900	1593	214	6.5
3000	1649	217	6.0

(a) Specimens annealed for one hour at pressures less than 1×10^{-5} torr (1×10^{-3} N/m^2).

(b) Average of four impressions at mid-wall location; 450-gram load.

(c) Intercept (or Heyn) procedure used.

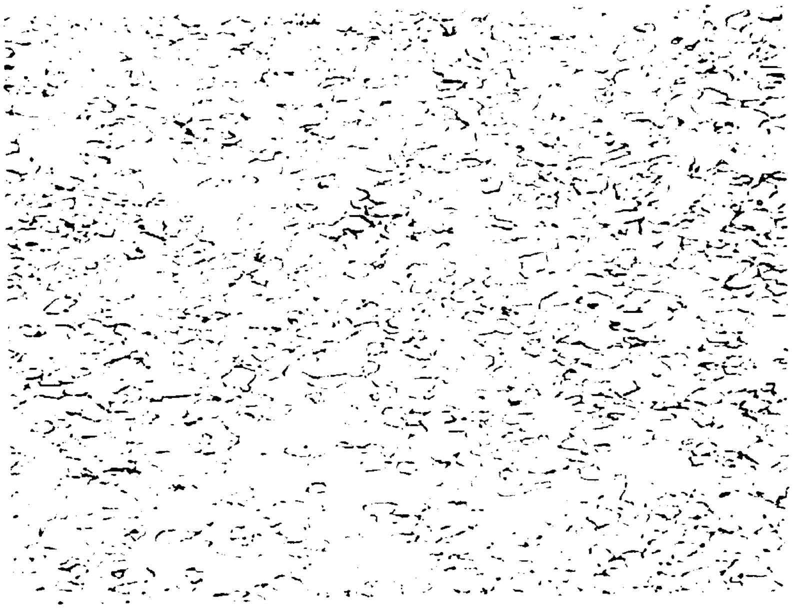
(d) No value, specimen heavily cold worked.

the base tubes were wrapped in tantalum foil and annealed for one hour at 2700°F (1482°C) at a pressure of less than 1×10^{-5} torr by Wolverine Tube.

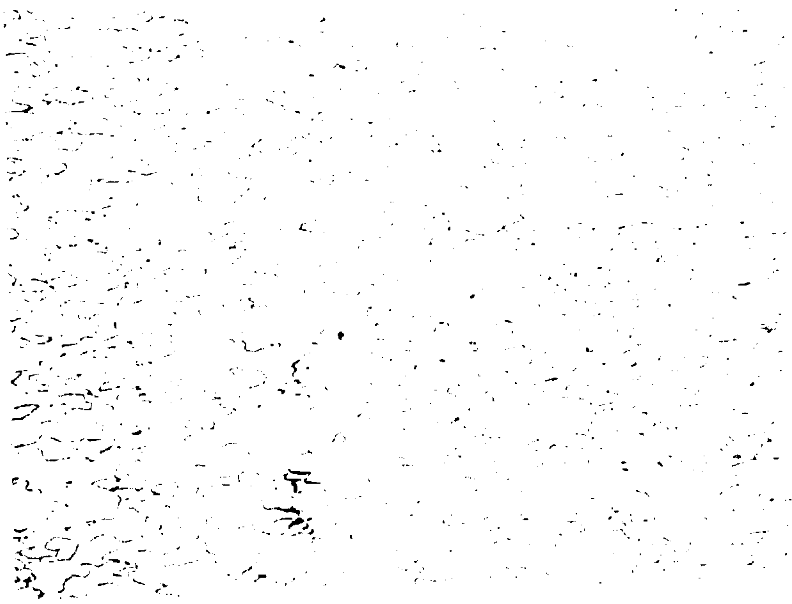
The base tubes were successfully given a 50 percent reduction to an intermediate tube size. Following this first tube reduction, a brief study was performed at General Electric to aid in selecting the annealing temperature to be used on the reduced tube prior to further tube reducing. Samples from the reduced tubes were annealed for one hour at 2400°, 2500°, 2600°, 2700°, 2800°, 2900°, and 3000°F (1316°, 1371°, 1427°, 1482°, 1538°, 1593°, and 1649°C) in vacuum. The samples were evaluated by means of metallographic examination and microhardness techniques.

Recovery and/or recrystallization have occurred in the annealed specimens shown in Figures D-9 through D-12. The microhardness and grain-size data are presented in Table D-IV. A one-hour anneal at 2700°F (1482°C) was selected to obtain a desirable combination of hardness and grain size. The tube was further reduced, annealing after each 50 percent reduction, to produce the 1.00-inch (2.5-cm)-OD x 0.10-inch (0.25-cm)-wall and 0.375-inch (0.95-cm)-OD x 0.065-inch (0.17-cm)-wall tubing. After cleaning and wrapping in tantalum foil, both the 1.00-inch (2.5-cm)-OD and 0.375-inch (0.95-cm)-OD tubing were given a final anneal for 1 hour at 3000°F (1649°C).

The final vendor inspection and testing of the T-111 alloy, 0.375-inch (0.95-cm)-OD x 0.065-inch (0.17-cm)-thick-wall and 1.0-inch (2.5-cm)-OD x 0.100-inch (0.25-cm)-thick wall tubing found the tubing to be free of rejectable defects. However, subsequent quality assurance inspection by ultrasonic testing at General Electric revealed numerous ID and OD defects in the 0.375-inch (0.95-cm)-OD tubing. Many of the defects were identified by visual examination, using a stereomicroscope, as OD defects of the scratch type which were apparently introduced during handling prior to receipt by General Electric. These defects were easily benched out. With the majority of the OD defects removed, the remaining ID defects were easier to identify in a second ultrasonic inspection. ID defects which exceeded the specification requirements were removed from the tube by sectioning. The metallographic appearance of typical ID defects of this type is shown in Figure D-13.



a) As Reduced

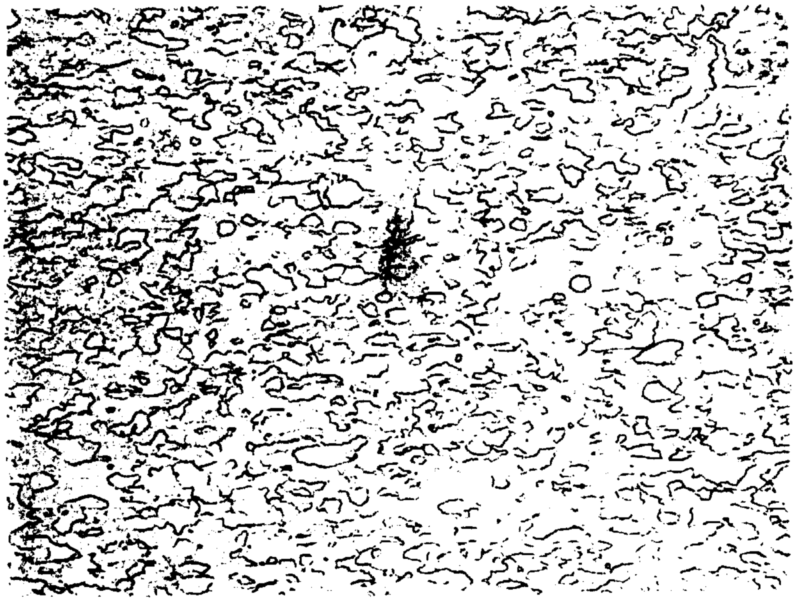


b) Annealed, 2400°F (1316°C) - 1 Hour C1127-1

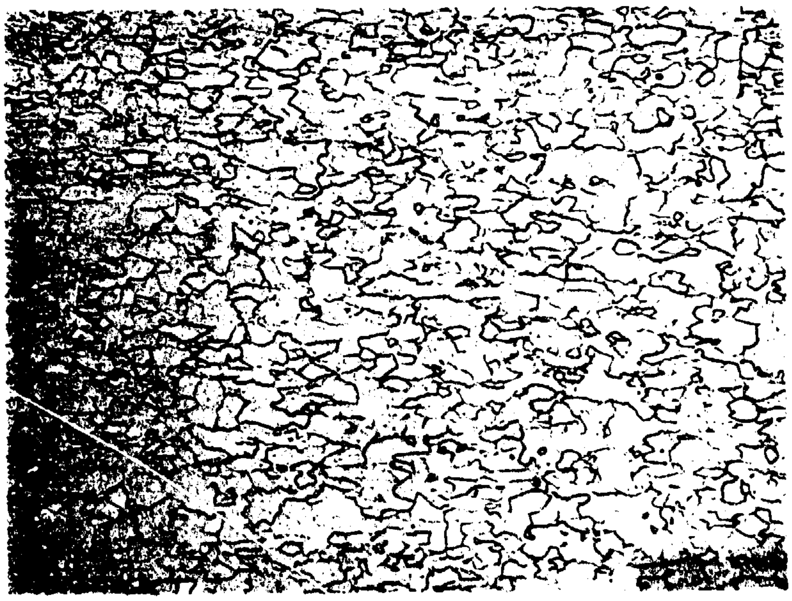
Figure D-9. Longitudinal Microstructure of T-111 Alloy Tubing After First Reduction.

Etchant: 30gmNH_4-20mlH_2O-50mlHNO_3

Mag.: 100X a) C100121, b) C100221



a) Annealed, :500°F (1371°C) - 1 Hour

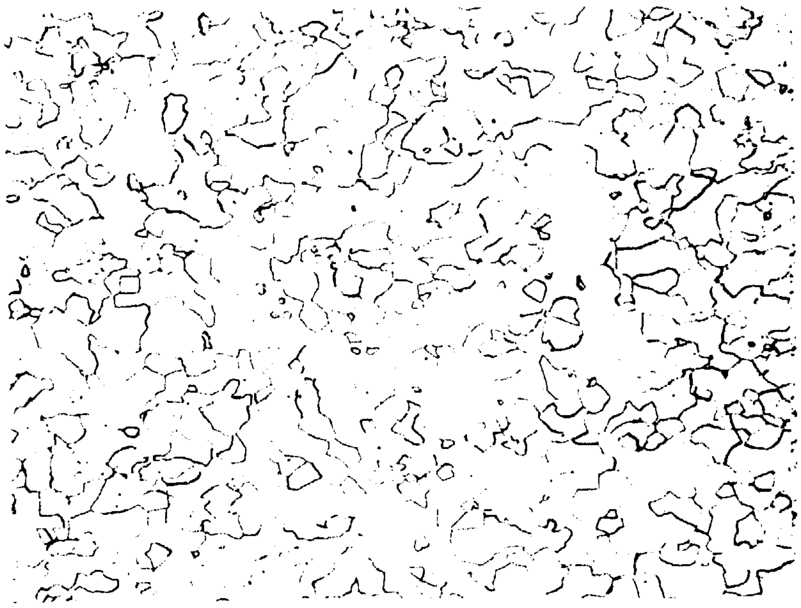


b) Annealed, 2600°F (1427°C) - 1 Hour C1127-2

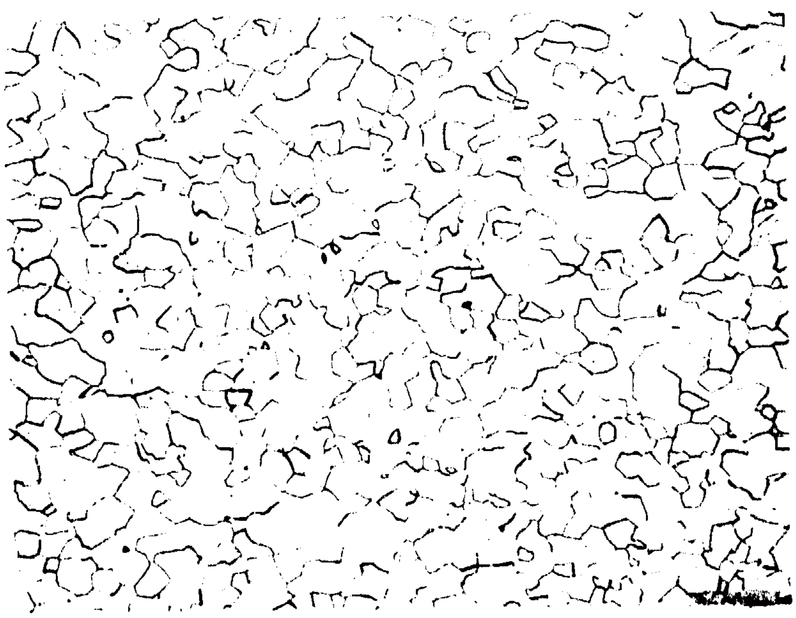
Figure L-10. Longitudinal Microstructure of T-111 Alloy Tubing After First Reduction.

Etchant: 30gmNH_4F-20mlH_2O-50mlHNO_3

Mag.: 100X a) C100321, b) C100421



a) Annealed, 2700°F (1482°C) - 1 Hour



b) Annealed, 2800°F (1538°C) - 1 Hour

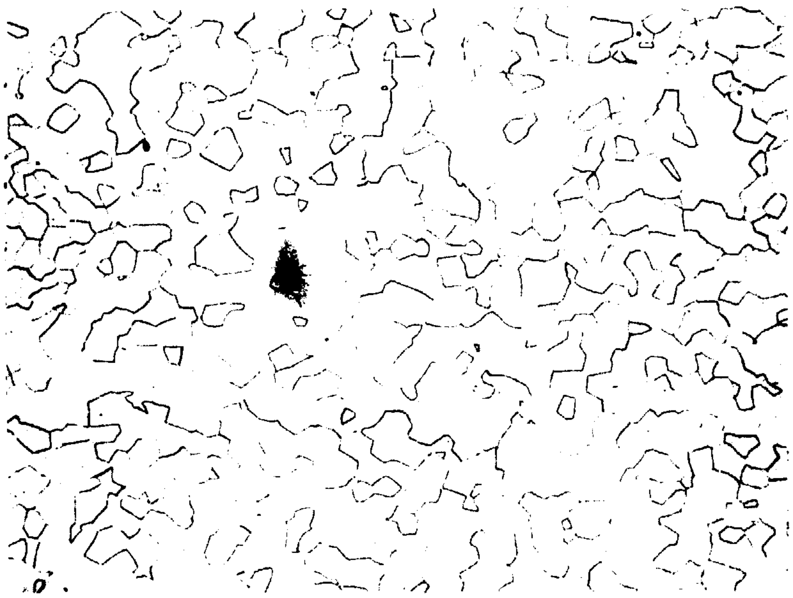
C1127-3

Figure D-11. Longitudinal Microstructure of T-111 Alloy Tubing After First Reduction.

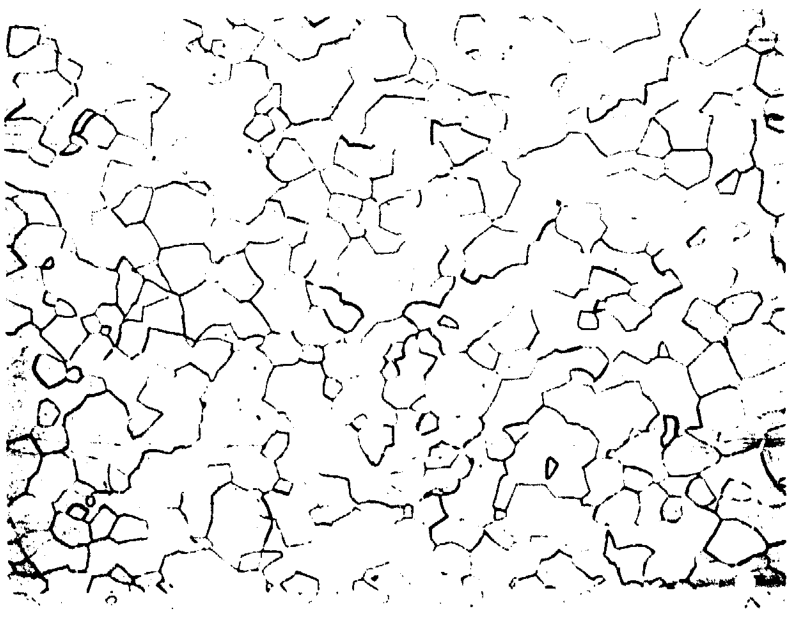
Etchant: 30gmNH_4F-20mlH_2O-50mlHNO_3

Mag.: 100X

a) C100521, b) C100621



a) Annealed, 2900°F (1593°C) - 1 Hour



b) Annealed, 3000°F (1649°C) - 1 Hour C1127-4

Figure D-12. Longitudinal Microstructure of T-111 Alloy Tubing After First Reduction

Etchant: 30gmNH_4F-20mlH_2O-50mlHNO_3

Mag.: 100X a) C100721, b) C100821

TABLE D-IV

MICROHARDNESS AND GRAIN SIZE OF UNANNEALED AND ANNEALED[(a)] T-111 ALLOY HEAT NUMBER 111-D-1670 AFTER FIRST REDUCTION

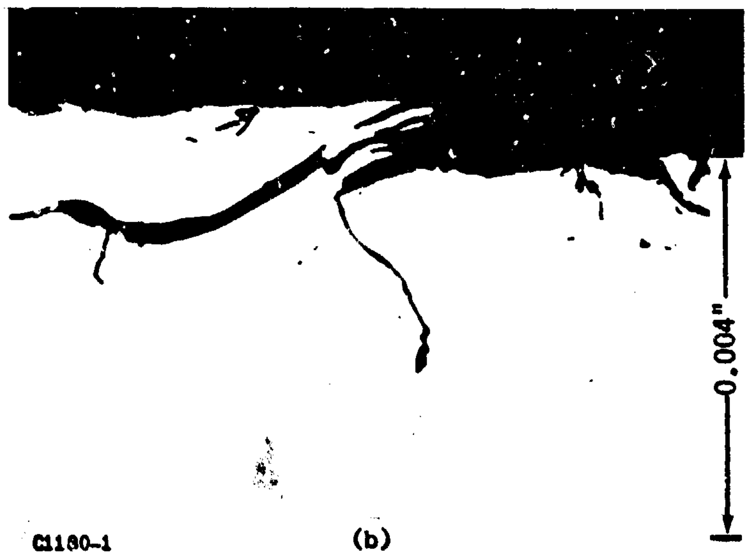
Annealing Temperature °F	Annealing Temperature °C	Microhardness[(b)] DPH	ASTM Grain Size[(c)]
As-Reduced		276	8.0
2400	1316	241	8.0
2500	1371	227	7.5
2600	1427	212	7.0
2700	1482	204	7.0
2800	1538	207	6.5
2900	1593	202	6.0
3000	1649	203	6.0

(a) Specimens annealed for one hour at pressures less than 1×10^{-5} torr.

(b) Average of four impressions at mid-wall location: 500-gram load.

(c) Intercept (or Heyn) procedure used.

(a)



C1180-1 (b)

Figure D-13. Photomicrographs of Typical Defects on the ID Surface and Inner Wall of 0.375-Inch (0.95-cm)-OD x 0.065-Inch (0.16-cm)-Wall T-111 Alloy Tubing.

Etchant: None a) C980223, b) C980222

Mag.: 500X

Heat No. 111D1102

After extrusion, the cans were removed, and the two billets were conditioned for rolling at Braeburn Alloy Steel Division of Continental-Copper and Steel Industries Incorporated. The conditioned 4.25-inch (10.8-cm)-diameter billets from the fourth ingot were rolled to 3.5-, 3.25-, 2.75-, and 2.25-inch (8.9-, 8.3-, 7.0-, and 5.7-cm) diameters at 1700°F (927°C) in air at Braeburn Alloy Steel. The finished products were given a final vacuum (10^{-5} torr [10^{-3} N/m^{2}]) anneal at 3000°F (1649°C) for 1 hour at the Boeing Airplane Company, Seattle, Washington.

Heat No. 111D1765

The fifth EB-melted Ta-8W alloy ingot (111D1765) was forged into 4.0-inch (10.2-cm)-diameter electrode stock at Anderson-Schumaker, Chicago, Illinois, and subsequently arc-melted to the T-111 composition at Fansteel. The resulting 7.5-inch (19.1-cm)-diameter arc-melted ingot was machined to 6.72 inches (17.1 cm), sectioned into two equal lengths, and shipped to Canton Drop Forging Company for extrusion. The two billets were canned in a molybdenum-lined Type 304 stainless steel seamless pipe by heliarc-welding. After extrusion, the cans were removed and the two billets were conditioned to 4-inch (10.2-cm) diameter and subsequently rolled to approximately 3-inch (7.6-cm) diameter at Braeburn Alloy Steel. The rolling was performed at 800°F (427°C) in air. The products made from the fifth T-111 alloy ingot were given a final vacuum-anneal at 3000°F (1649°C) for one hour at the Boeing Company, Seattle, Washington.

WAH CHANG ALBANY DIVISION

A portion of the T-111 alloy mill products required for the program was produced by Wah Chang. Consolidation of the T-111 alloy ingots is similar to the process described for the ingots produced by Fansteel. Blended tantalum and tungsten pwoders are compacted, sintered, and double electron-beam-melted to product a Ta-8W alloy electrode for arc melting. Hafnium in the form of crystal bar strip or electron-beam-melted strip is GTA-welded along the entire length of the electrode. The electrode then is double vacuum-arc-melted to form a 6 1/2-inch (16.5-cm)-diameter ingot.

Primary conversion of the conditional double arc-melted ingot is accomplished by forging at temperatures on the order of 2400°F (1316°C). A proprietary coating is applied to the ingot to minimize contamination during processing. After initial breakdown of the ingot, subsequent processing to finish sizes is accomplished at relatively low temperatures using standard facilities and techniques. Final annealing of the mill products was carried out at 3000°F (1649°C)/1 hour in vacuum. Material produced by Wah Chang was foil and sheet in thicknesses of 0.005, 0.009, 0.035, 0.125, and 0.050 inch (0.013, 0.023, 0.089, 0.318, and 0.13 cm); rod in diameters of 0.062, 0.094, 0.125, 0.250, 0.500, 0.625, 1.00, 1.125, and 1.500 inches (0.157, 0.238, 0.317, 0.635, 1.270, 1.587, 2.54, 2.857, and 3.810 cm); bar of dimensions 1.0 x 1.0 inch (2.54 x 2.54 cm).

C.2

APPENDIX E

PROCESSING OF Mo-TZC ALLOY

CLIMAX MOLYBDENUM COMPANY OF MICHIGAN

Consolidation of the Mo-TZC alloy (Mo-1.25Ti-0.15Zr-0.15C) at Climax was performed in their PSM facility. The blended powders are compressed, sintered to form an electrode, and consumably vacuum arc melted in one operation. One 9-inch (22.9-cm)-diameter ingot was melted to fulfill the program requirements. The cropped and trimmed ingot, measuring 6 15/16 inches (17.6 cm) in diameter was vacuum annealed at 2800°F (1538°C) for 1 hour. One billet cut from the ingot was canned in unalloyed molybdenum by heliarc welding and shipped to Allegheny Ludlum, Watervliet, New York, for extrusion; the diameter of the canned ingot was 7 1/8 inches (18.1 cm). The ingot was heated by induction to 3200°F (1760°C) (optical) in an argon atmosphere. An attempt to extrude the Mo-TZC alloy ingot in a 7 3/8-inch (18.7-cm)-ID container was unsuccessful as failure of a valve to the accumulator resulted in a loss of pressure. Severe cracking of the ingot occurred as a result of the upsetting action in the container.

Two additional billets of Mo-TZC, machined from the same heat, were shipped to Allegheny Ludlum for extrusion. Prior to shipment, both ingots were vacuum-annealed for one hour at 2800°F (1538°C). The ingot sizes were:

Diameter	Length	Weight
5.4 in. (13.7 cm)	12 in. (30.5 cm)	102 lb (46.3 kg)
6.0 in. (15.2 cm)	21 in. (53.3 cm)	233 lb (106 kg)

The 5.4-inch (13.7-cm)-diameter ingot was machined from the bottom of the original ingot and required more cleanup of the surface. The 21-inch (53.3-cm) length is a limiting dimension because of the length of the induction coil used for heating the ingots.

A second attempt to extrude at Allegheny Ludlum with the 5.4-inch (13.7-cm) ingot also was unsuccessful. The ingot failed to extrude at the full capacity of the press for the type and size of stem that was used. Examination of the billet showed that about 2 inches (5 cm) had extruded

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before the ingot stalled in the press, and it was apparent that very little of the lubricant was on the surface. It is believed that lack of lubrication due to the possible use of too high a melting glass and/or the flat, shear-type die were responsible for the failure of the ingot to extrude. Subsequent inspection of the ingot revealed that severe cracking had occurred from the upsetting action.

The remaining 6-inch (15.2-cm)-diameter, Mo-TZC alloy billet was turned to 5.85-inch (14.9-cm) diameter for extrusion at duPont. The ingot was induction heated to 3180°F (1749°C) in argon atmosphere and extruded through a 3-inch (7.6-cm)-ID, ZrO_2-coated conical die. The extrusion parameters are shown in Table E-I. As the result of severe die wash, the extrusion diameter was close to 3 1/8 inches (7.9 cm). Also, there appeared to be a loss of lubrication on one side of the extrusion which resulted in a series of transverse surface cracks along the unlubricated surface. These cracks were successfully removed during subsequent conditioning of the billet for rolling at Hoskins Corporation, Detroit, Michigan.

The results of recrystallization studies of longitudinal samples from the midradius of the extruded billet are summarized in Table E-II. Although an annealing temperature of 3400°F (1871°C) resulted in an essentially recrystallized structure, metallographic examination revealed continuous films of Mo_2C at the grain boundaries. Similarly, the sample which was heated at 3200°F (1760°C) for one hour also exhibited some Mo_2C in the grain boundaries. Since the presence of Mo_2C in the grain boundaries is not a desirable condition for subsequent working operations, the postextrusion heat-treatment was limited to a stress-relief anneal at 3000°F (1649°C) for one hour in vacuum. A solution anneal at temperatures on the order of 3750°V (2066°C) was not employed because of possible grain growth and the fact that only a 52 percent reduction is required to reach the final size of 2 inches (5 cm) in diameter.

The conditioned and stress-relieved Mo-TZC alloy billet was successfully rolled by Hoskins Corporation, Detroit, Michigan, to 2 1/8-inch (5.4-cm)-diameter rod. A rolling temperature of 2400°F (1316°C) was utilized. The 2-inch (5-cm)-diameter rod was machined from a portion of the rolled 2 1/8-inch (5.4-cm)-diameter rod. The remaining 2 1/8-inch (5.4-cm)-diameter rod

TABLE E-I

EXTRUSION PARAMETERS FOR THE 5.85-INCH (14.9-cm)-DIAMETER MACHINED Mo-TZC ALLOY INGOT[a]

Billet Size	-	5.85-inch (14.9-cm) diameter
Container Size	-	6.0-inch (15.2-cm) ID
Die Size/Design	-	3.0-inch (7.6-cm) ID/90° conical
Die Coating	-	ZrO_2
Extrusion Ratio	-	3.8/1
Lubricant	-	Billet coated with 7052 glass; glass lubricated with Fiske grease. Die lubricated with 7052 glass powder and a glass-wool pad.
Billet Temperature	-	3180°F (1749°C) (induction-heated in argon)
Extrusion pressure	-	126,000 psi (868 MN/m^2) 115,500 psi (796 MN/m^2) run-out
Cooling Procedure	-	Air-cooled to 1700°F (927°C); cooled in Sil-O-Cel from 1700°F (927°C) to room temperature.

(a) Extruded at E.I. duPont de Nemours and Company, Inc., Baltimore, Maryland.

TABLE E-II

RECRYSTALLIZATION[a] OF 3.125-INCH (8.0-cm)-DIAMETER Mo-TZC ALLOY EXTRUSION[b]

Temperature[c] °F	°C	Vickers' Hardness Number	% Recrystallization
As extruded		232	10
2800	1538	240	15
3000	1649	236	15
3200	1760	229	25
3400	1871	201	98

(a) Conducted by the Climax Molybdenum Company of Michigan, Ann Arbor, Michigan.

(b) Extruded at E. I. duPont de Nemours and Company, Inc., Baltimore, Maryland, in 6.0-inch (15.2-cm)-OD container - ingot diameter 5.85 inches (14.9 cm).

(c) Temperatures maintained for one hour in vacuum.

was rolled at 2400°F (1316°C) to 1.3-inch (3.3-cm)-diameter rod by Climax Molybdenum Company, Coldwater, Michigan. The rod was subsequently machined to 1-inch (2.5-cm) diameter. The chemical analyses of the ingot and stress-relieved rod, as supplied by Climax, are shown in Table E-III. The mechanical properties, also supplied by Climax, are presented in Table E-IV.

GE-LMCD

Four six-inch (15.2-cm)-diameter, Mo-TZC alloy ingots were single vacuum-arc-melted by the Lamp Metals and Components Department of General Electric Company. The ingots were machined to a nominal 5 1/16-inch (12.9-cm) diameter and extruded at the GE-R&D Center, Schenectady, New York. The extrusion parameters that were employed are shown in Table E-V.

Ultrasonic inspection of all of the extrusions revealed no internal defects. One ingot (Heat No. 4454) stuck during extrusion but it was possible to remachine the surface and successfully reextrude to bar.

Two extrusions (4451 and 4453) were cross rolled to 1 3/8-inch (3.5-cm)-thick plate for subsequent machining into nozzles for the turbine simulator. Attempts made to roll the first extrusion (4451) at a temperature of 2390°F (1300°C) resulted in severe cracking. The rolling cycle was modified for the second extrusion (4453) to provide for a 20 percent reduction to be made at 2912°F (1600°C) and a 20 percent reduction to be made at 2390°F (1300°C). The material was rolled to this schedule without difficulty.

The third extrusion (4454) was successfully cross rolled to 3/4-inch (1.91-cm)-thick plate with approximately 40 percent reduction being made at a temperature of 2912°F (1600°C) and approximately 40 percent reduction being made at a temperature of 2390°F (1300°C). This material is to be utilized for the machining of turbine blades. The chemical analyses of the rolled plate, as supplied by GE-LMCD, are shown in Table E-VI, and the mechanical properties are presented in Table E-VII.

DETERMINATION OF FINAL ANNEALING TREATMENT

Because of the marginal room temperature ductility of the heavy section

TABLE E-III

CHEMICAL ANALYSES OF Mo-TZC ALLOY (HEAT NO. TZC-4431) PRODUCED BY CLIMAX MOLYBDENUM COMPANY

		Chemical Analyses					
		%			ppm		
Material	Sample Location	C[(a)]	Ti[(b)]	Zr[(b)]	O[(c)]	H[(c)]	N[(c)]
Ingot	Top - center	0.13	1.21	0.18			
	Top - mid-radius	0.13	1.20	0.14			
	Top - edge	0.13	1.21	0.21			
	Bottom - center	0.13	1.19	0.15			
2-inch (5.1-cm)-diameter rod	Cross section	0.13			4	< 1	2
1-inch (2.54-cm)-diameter rod	Cross section	0.13			4	< 1	< 1

(a) Combustion analysis.

(b) X-ray fluorescence analysis.

(c) Vacuum fusion analysis.

TABLE E-IV

MECHANICAL PROPERTIES[(a)] OF Mo-TZC ALLOY (HEAT NO. TZC-441) PRODUCED BY CLIMAX MOLYBDENUM COMPANY

Specimen Location	Room Temperature Tensile Properties[(b)]: Ultimate Strength 1000 psi	0.2% Yield Strength 1000 psi	Elong. %	Stress-Rupture Life at 2400°F (1316°C) & 30,000 psi in Vacuum of 3×10^{-5} Torr[(c)]: Life Hours	Minimum Creep Rate %/Hour	Elong. %	Reduction in Area %
1-inch (2.54-cm)-diameter bar, mid-radius	118.4	106.0	15.5	35.5	0.052	28	86.1
	118.0	109.1	32.5	30.2	0.043	30	86.4
2-inch (5.1-cm)-diameter, bar mid-radius	104.1	96.1	1.5	73.2	0.03	33	64
	106.1	97.1	2.0	101.9	0.03	29	79

(a) Tensile and stress-rupture specimens were machined from Mo-TZC alloy bar following annealing for one hour at 2400°F (1316°C). The axes of the specimens were parallel to the rolling direction. The specimens had a 0.25-inch (0.635-cm) diameter with a 1.250-inch (3.18-cm)-gauge length and were hand-polished prior to testing.

(b) Tensile properties were determined using a strain rate of 0.005 inch/inch/min. up to 0.6% offset and then 0.050 inch/inch/min. to fracture.

(c) Stress-rupture specimens were held for a half-hour at 2400°F (1316°C) before load was applied.

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TABLE E-V

EXTRUSION PARAMETERS FOR Mo-TZC ALLOY INGOTS[(a)]

Machined Ingot Size	-	5.06-inch (12.9-cm) diameter
Leader Block	-	None
Follow-up Block	-	Graphite
Container Size	-	5.187-inch (13.1-cm) ID
Die Size/Design	-	2.1-inch (5.35-cm) x 4.1-inch (10.4-cm)/ conical
Die Coating	-	ZrO_2
Lubricant - Proprietary	-	Ugine'-Sejournet processed not used

		Heat No.		
		4451	4454	4453
Extrusion Ratio	-	2.29/1	2.24/1	--
Furnace Temperature, °F	-	3110	3110	3092
Furnace Temperature, °C	-	1710	1710	1700
Soak Time, Hours/Minutes in Hydrogen	-	3:25	3:58	3:54
Handling Time, Seconds	-	22.7	24.3	22.1

(a) Extruded at General Electric Research and Development Center, Schenectady, New York.

TABLE E-VI

CHEMICAL ANALYSES[a] OF Mo-TZC ALLOY PRODUCED BY GENERAL ELECTRIC-LMCD

Chemical Analysis	Heat No. 4451 (M92)[b]		Heat No. 4453 (M97)[b]		Heat No. 4454[c]	
	Nose[d]	Tail	Nose	Tail	Nose	Tail
Ti	1.4 %	1.3 %	1.4 %	1.3 %	1.4 %	1.4 %
Zr	0.20%	0.19%	0.20%	0.17%	0.21%	0.21%
O	2 ppm	4 ppm	5 ppm	5 ppm	5 ppm	7 ppm
N	3	3	2	2	2	3
H	2	1	2	2	2	2
C	1172	1169	1205	1120	1314	1303
Al	<8	<8	<8	<8	<8	16
Ca	<5	<5	<5	<5	<5	11
Si	48	22	27	27	15	23
Cr	9	<8	11	10	15	10
Fe	47	47	35	19	35	19
Ni	<5	<5	<5	6	9	7
Cu	4	4	7	8	5	<4
W	<150	<150	<150	<150	100	136
Mn	10	10	12	12	18	13
Mg	<10	<10	<10	<10	16	<10
Sn	<7	<7	12	13	10	<7
Co	<20	<20	<20	<20	<20	<20
Pb	<10	<10	<10	<10	<10	<10
Ta	<100	<100	<100	<100	<100	<100
Cb	<100	<100	<100	<100	<100	<100
V	<50	<50	<50	<50	<50	<50
Ag	<1	<1	<1	<1	<1	<1

[a] Analyses performed by GE-LMCD. Ti and Zr determined by wet chemistry. C determined by conductometric techniques. O, N, H determined by vacuum fusion. Other metallics analyzed by spectrographic techniques.

[b] Samples obtained from rolled 1 3/8-inch (3.5-cm)-thick plate.

[c] Samples obtained from rolled 3/4 inch (1.91-cm)-thick plate.

[d] Indicates location of chemistry sample with respect to extrusion.

TABLE E-VII

MECHANICAL PROPERTIES OF Mo-TZC ALLOY PRODUCED BY GENERAL ELECTRIC-LMCD

Heat No.	Specimen Location[c]	Room Temperature Tensile Properties[d]: Ultimate Strength, 1000 psi	0.2% Yield Strength, 1000 psi	Elongation %	Stress-Rupture Life[e] Vacuum (10^{-6} Torr) at 2400°F/30,000 psi: Life Hours	Elongation %
4451[a] (M92)	Nose[f]	134.6	105.9	1.3	37.81	12.4
	Tail[g]	126.5	105.0	1.3	20.94	13.3
4453[a] (M97)	Nose[h]	96.6	95.2	0.4	28.57	2.2
	Tail[h]	94.7	92.8	0.3	21.88	3.5
4454[b]	Nose[i]	111.3	101.9	2.1	19.33	13.4
	Tail[i]	95.9	94.5	0.4	---	--

[a]Specimens obtained from rolled 1-3/8-inch thick plate and tested by GE-LMCD.

[b]Specimens obtained from rolled 3/4-inch thick plate and tested by GE-LMCD.

[c]Indicates location of mechanical property sample with respect to the extrusion.

[d]Specimens were 0.250-inch diameter with 1.0-inch gauge length. Strain rate 0.005-inch/inch/minute.

[e]Specimens were 0.159-inch diameter with 1.0-inch gauge length.

[f]Tensile specimen axis perpendicular to rolling direction and specimen annealed for 1 hour at 2192°F before testing; stress-rupture specimen axis parallel to rolling direction and specimen annealed 1 hour at 2372°F before testing.

[g]Tensile specimen axis perpendicular to rolling direction and stress-rupture specimen axis parallel to rolling direction. Both specimens were annealed 1 hour at 2372°F before testing.

[h]Tensile and stress-rupture specimen axis parallel to rolling direction and all specimens annealed 1 hour at 2372°F before testing.

[i]Tensile and stress-rupture specimen axis parallel to rolling direction of 3/4-inch-thick plate and specimens annealed 1 hour at 2372°F before testing.

sizes of the Mo-TZC alloy products, a heat-treat study was performed on the Mo-TZC alloy rod to define an annealing treatment which will improve the room-temperature ductility without appreciable recrystallization or reduction in the yield strength.

The Mo-TZC alloy mill products manufactured by the two vendors (General Electric-LMCD and Climax Molybdenum Company) were processed using different working schedules. Therefore, a single optimum heat treatment for all the Mo-TZC alloy mill products could not be selected. Results of this study resulted in the following courses of action:

a. The 0.75-inch (1.91-cm)-thick plate (GE-LMCD) and the 2.0-inch (5.1-cm)-diameter rod (Climax) were heat treated for 50 hours at 2600°F (1427°C).

b. The 1.375-inch (3.5-cm)-thick plate (GE-LMCD) was not used in the Corrosion Loop Program because of its nonuniform microstructure.

c. The 1-inch (2.5-cm)-diameter rod (Climax) was utilized in the as-received condition (1 hour at 2400°F (1316°C).

The heat-treatment study was conducted with approximately 0.25-inch (0.64-cm)-thick slices cut from the ends of each of the Mo-TZC alloy mill shapes. The slices from the plate material were cut into cubes, and the slices from the rods were cut into pie-shaped specimens. After chemical cleaning, the specimens were heat treated for one hour at 2600°, 2700°, 2800°, 3000°, 3200°, 3400°, and 3750°F (1427°, 1482°, 1538°, 1649°, 1760°, 1871°, and 2066°C) in a vacuum of less than 1×10^{-5} torr and subsequently quenched in helium.

The microstructures and microhardnesses of the 0.75-inch (1.9-cm)-thick Mo-TZC alloy before and after the one-hour heat treatment are shown in Figure E-1. Significant recrystallization was observed in the specimen heat treated for one hour at 2600°F (1427°C) as well as a low hardness. The data obtained for this particular heat treatment are considered anomalous with respect to the data obtained after performing other heat treatments on this material. The one-hour heat treatment at 2800°F (1538°C) is considered to be the condition in which significant recrystallization will occur in the 0.75-inch (1.9-cm)-thick plate. Also, the microhardness data indicate a rapid decrease in hardness after a one-hour heat treatment at a temperature

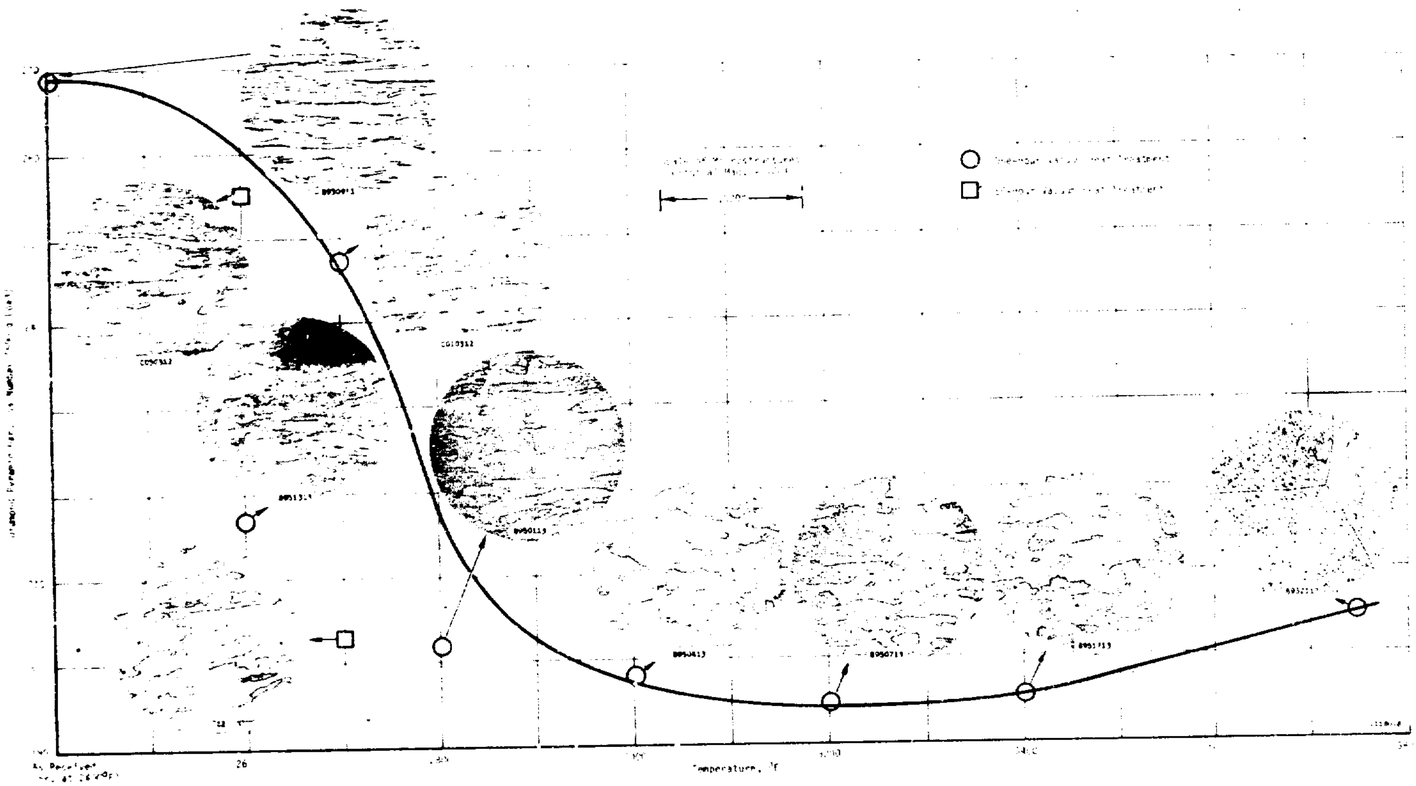

Figure E-1. Microhardness and Microstructure of Longitudinal Specimens Cut From 0.75-Inch (1.9-cm)-Thick Mo-TZC Alloy Plate as a Function of Heat-Treatment Temperature. Etchant: 50% Murakamis of 2700°F (1482°C). From the metallographic and microhardness data, a temperature of 2700°F (1482°C) or less was suggested for a one-hour heat treatment of the 0.75-inch (1.9-cm)-thick plate.

The effects of longer time heat treatments on microstructure and hardness of the 0.75-inch (1.9-cm)-thick plate were evaluated by heat treating samples for 50 hours at 2600°F and 2700°F (1427°C and 1482°C). The microstructures, Figure E-1, indicate only a slight amount of recrystallization in the specimen heat-treated at 2600°F (1427°C) for 50 hours and a significant amount of recrystallization in the specimen heat treated at 2700°F (1482°C) for 50 hours. The microhardness data, Figure E-1, are in agreement with the microstructures in that the hardness of the specimen heat treated at 2700°F (1482°C) was considerably reduced. These results indicate that a 50-hour heat treatment at 2600°F (1427°C) also may be beneficial in achieving greater tensile ductility at the lower temperatures. A longer-time heat treatment has the advantage of allowing more time for carbon precipitation.

The microstructures and microhardness of the 1.375-inch (3.49-cm)-thick Mo-TZC alloy plate before and after the one-hour heat-treatments are shown in Figure E-2. Significant recrystallization was observed after heat treating for one hour at 3200°F (1760°C). This high recrystallization temperature is attributed to the relatively small amount of cold-work that this material received during processing, as shown in the as-received microstructure which is noticeably less uniform than the microstructure of the other materials. In general, the microstructure data for the one-hour heat treatments below 3200°F (1760°C) indicate that recovery is the major process taking place with very small localized areas, which received a greater amount of effective cold-work, starting to recrystallize. Since the slope of the hardness curve is fairly uniform up to 3200°F (1760°C) and microstructural changes are slight, it would be desirable to maintain the temperature of a one-hour heat treatment for the 1.375-inch (3.49-cm)-thick Mo-TZC alloy as low as possible to reduce degradation of strength and high enough to precipitate carbon from solution in a reasonable time period. The effects of longer-time heat treatments on the 1.375-inch (3.49-cm)-thick plate were also evaluated by heat

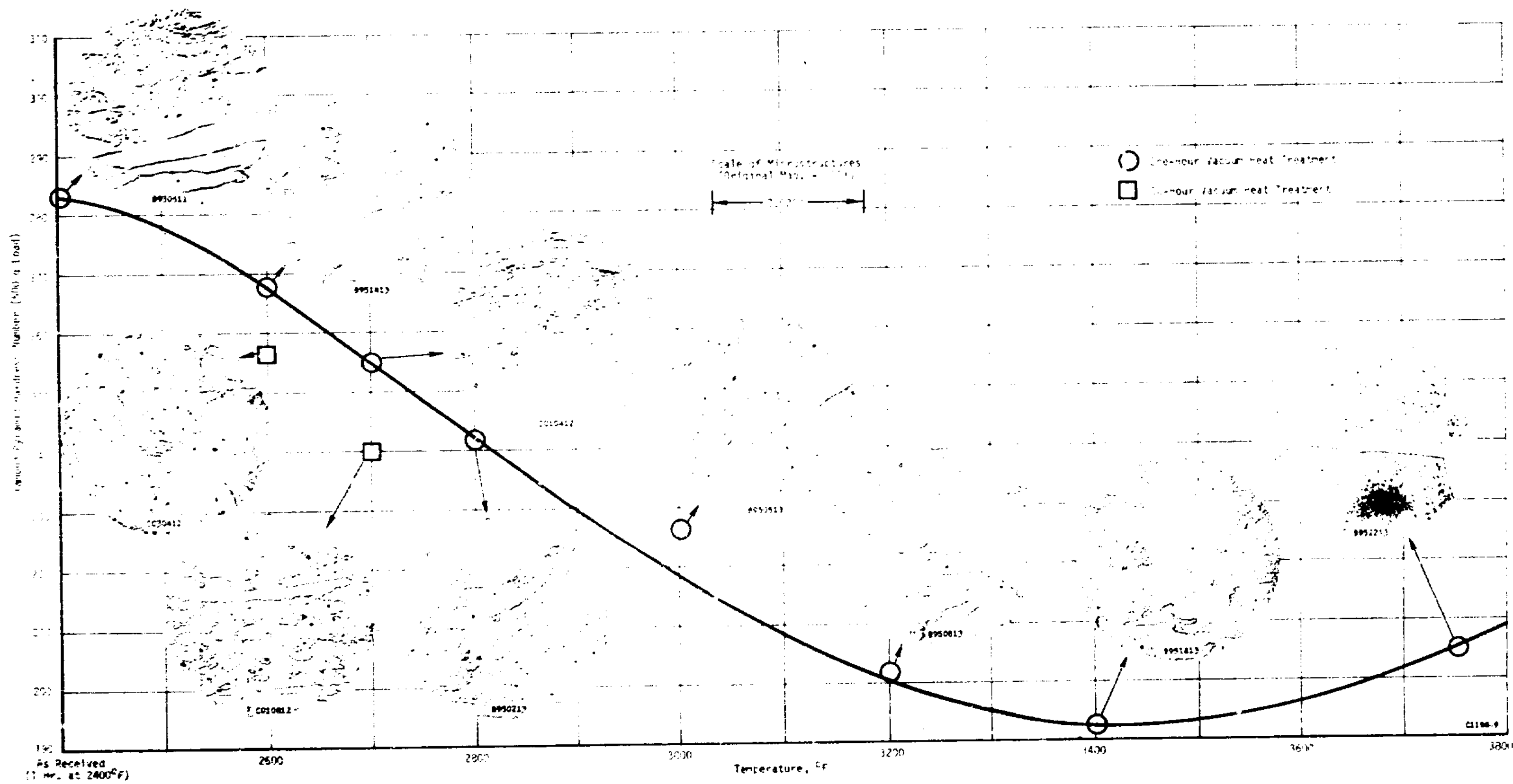

Figure E-2. Microhardness and Microstructure of Longitudinal Specimens Cut From 1.375-Inch (3.49-cm)-Thick Mo-TZC Alloy Plate as a Function of Heat-Treatment Temperature. Etchant: 50% Murakamis treating samples for 50 hours at 2600°F and 2700°F (1427°C and 1482°C). The microstructures of these specimens are shown in Figure E-2 and indicate that recovery is predominant with some recrystallization in small localized areas in the specimen heat treated for 50 hours at 2600°F (1427°C) and a significant amount of recrystallization in the specimen heat treated at 2700°F (1482°C) for 50 hours. A decrease in hardness was observed in both specimens, Figure E-2, with the hardness values being slightly less than the one-hour heat treatments at 2600°F (1427°C) and 2700°F (1482°C). These data indicate that a 50-hour heat treatment at 2600°F (1427°C) would be the preferred heat treatment to improve the low-temperature tensile ductility of the 1.375-inch (3.49-cm)-thick plate with a minimal reduction in yield strength.

The room-temperature tensile ductility of the 1.0-inch (2.5-cm)-diameter, Mo-TZC alloy rod produced by Climax Molybdenum is satisfactory for use in the as-received condition (2400°F [1316°C]/1 hour). However, heat-treatment studies were conducted with this material to determine its recrystallization characteristics. The microstructures and microhardness after the one-hour heat treatments are shown in Figure E-3. Significant recrystallization was observed in the specimen which was heat treated at 3000°F (1649°C) for one hour. The microhardness data shown a rapid decrease in hardness after one hour at 2800°F (1538°C). The effects of longer-time heat treatments (50 hours at 2600° and 2700°F, 1427° and 1482°C) on the 1.0-inch (2.5-cm)-diameter material are shown in Figure E-3. Metallographic examination revealed the initiation of recrystallization in both heat treatments but to an insignificant amount. A slight decrease in microhardness also was observed.

Specimens from the 2.0-inch (5.1-cm)-diameter Mo-TZC alloy rod produced by Climax Molybdenum were heat treated for one hour at 2800°F (1538°C) and 3000°F (1649°C). Significant recrystallization was observed after heat treating for one hour at 3000°F (1649°C) and a slight decrease in microhardness was noted after the one-hour heat treatment at 2800°F (1538°C). These results indicate that a one-hour heat treatment to precipitate carbon from solution should be carried out at a temperature of 2800°F (1538°C) or less. Additional specimens from the 2.0-inch

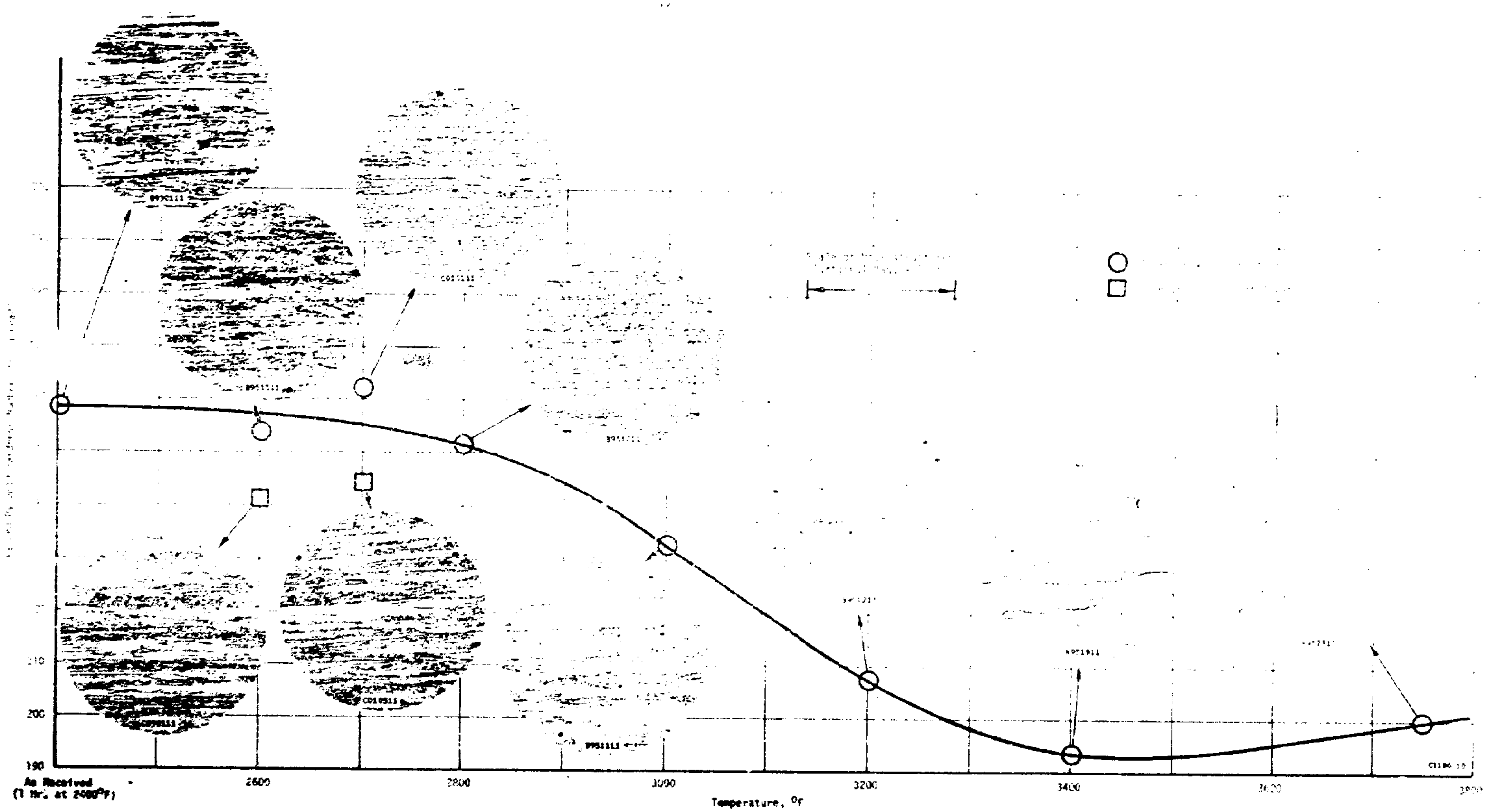

Figure E-3. Microhardness and Microstructure of Longitudinal Specimens Cut From 1.0-Inch (2.5-cm)-Diameter Mo-TZC Alloy Rod as a Function of Heat-Treatment Temperature. Etchant: 50% Murakamis

(5.1-cm)-diameter rod were heat treated for 50 hours at 2600°F (1427°C) and 2700°F (1482°C). Significant recrystallization was observed in the specimen heat treated at 2700°F (1482°C) and is reflected in the low microhardness of the specimen. The specimen heat treated at 2600°F (1427°C) for 50 hours exhibited a very small amount of recrystallization and a slight decrease in hardness. These data indicate that a 50-hour exposure at 2600°F (1427°C) could be used for the final heat treatment of the 2.0-inch (5.0-cm)-diameter material.

As a result of these studies, 50 hours at 2600°F (1427°C) was selected as a promising heat treatment for the comparison of tensile properties with the material in the as-received condition of one hour at 2400°F (1316°C). Buttonhead tensile specimens, Figure E-4, were machined from Mo-TZC rod and bar such that the longitudinal axes of the specimens were parallel to the rolling direction. The finished tensile specimens were chemically cleaned and carefully inspected for surface defects using fluorescent penetrant techniques. The tensile specimens were vacuum heat treated at pressures less than 1×10^{-5} torr after the machining, cleaning, and inspection operations. The tensile tests were conducted in air at room temperature to 400°F (204°C) and were performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." A strain rate of 0.005 inch/inch/minute up to 0.6 percent offset and then 0.050 inch/inch/minute to fracture was used; the yield strength was determined by the 0.02 percent and 0.2 percent offset methods. The results of these tests along with the data reported by the vendors are shown in Table E-VIII.

In general, an increase in room-temperature ductility and a decrease in room-temperature yield strength were observed in the specimens tested from all four mill shapes which were heat treated for 50 hours at 2600°F (1427°C). It was concluded that the improved ductility was more significant to the utilization of these materials in the T-111 Corrosion Loop than the observed decrease in strength except for the 1.0-inch (2.5-cm)-diameter rod material. The 1.0-inch (2.5-cm)-diameter Mo-TZC alloy rod exhibited very good ductility in the as-received condition (heat treated for one hour at 2400°F [1316°C]), and the slight increase observed after heat treating

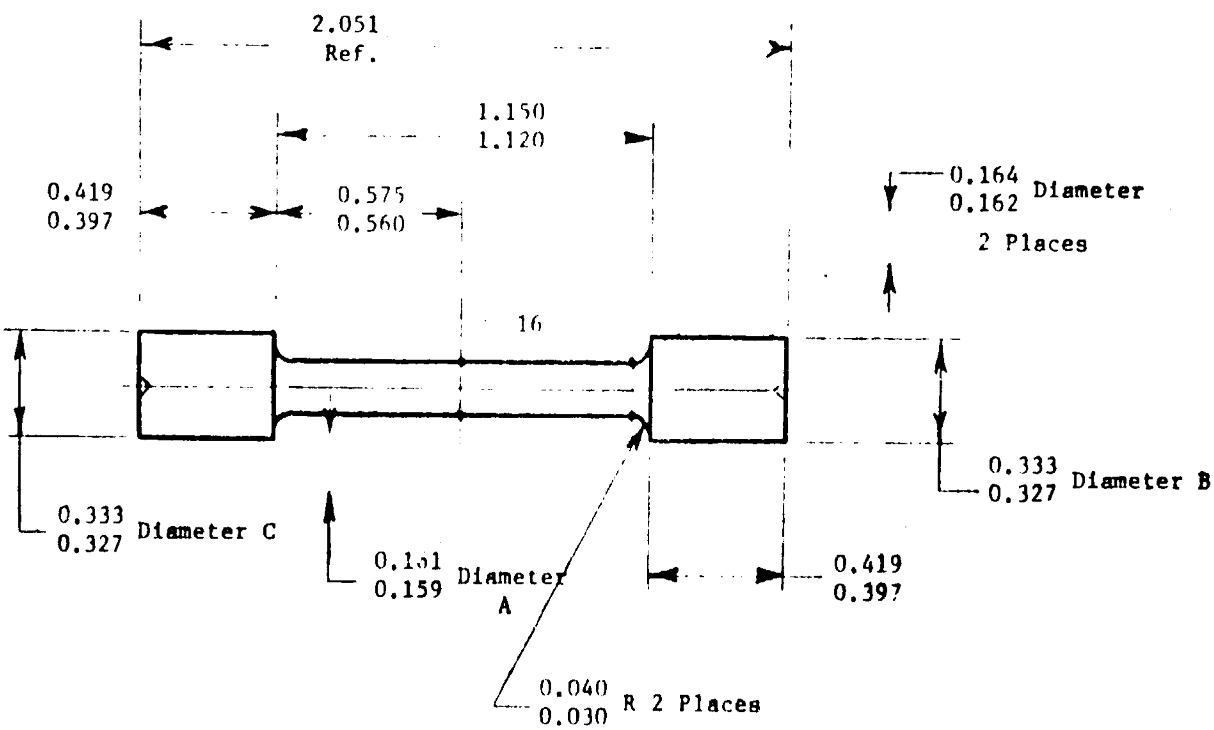

Figure E-4. Design of Buttonhead Tensile Specimens Machined From Cb-132M Alloy and Mo-TZC Alloy Materials

TABLE E-VIII

TENSILE PROPERTIES FOR Mo-TZC ALLOY PLATE(a) AND ROD(b)

Material	Specimen No.(b)	Test Temp °F	Test Temp °C	Ultimate Tensile Strength psi x 10^{-3}	0.02% Yield Strength(c) psi x 10^{-3}	0.2% Yield Strength(c) psi x 10^{-3}	Reduction in Area (%)	Elong. % in 1 Inch
0.75-Inch (1.9-cm)-Thick Plate	(d)	78	26	111.3	Not Reported	101.9	--	2.1
	2	150	66	111.0	72.0	86.0	11.5	9.5
	1	225	107	107.0	72.5	82.5	11.0(e)	8.0
	3 (f)	78	26	106.0	67.1	75.0	9.0	7.5
	4 (f)	150	66	88.1	53.3	61.0	28.5	21.0
1.375-Inch (3.5-cm)-Thick Plate	(d)	78	26	94.7	Not Reported	92.8	--	0.3
	(d)	78	26	96.6	Not Reported	95.2	--	0.4
	21	225	107	90.0	65.2	73.2	2.5	3.0
	25	300	149	99.0	73.0	80.0	4.0	2.5
	22	400	204	88.7	62.4	70.9	51.5	15.5
	23 (f)	81	27	96.5	70.3	80.0	1.0(g)	3.5
	24 (f)	150	66	98.5	52.0	72.3	12.0	12.0
1.0-Inch (2.54-cm)-Diameter Rod	(d)	78	26	118.4	--	106.0	--	15.5
	(d)	78	26	118.0	--	109.1	--	32.5
	29	88	31	114.0	81.0	97.8	53.5	25.0
	30	88	31	117.2	82.8	101.0	38.0	20.0
	31 (f)	80	27	107.0	80.8	89.0	38.0(h)	26.0
2.0-Inch (5.1-cm)-Diameter Rod	(d)	78	26	104.1	--	96.1	--	1.5
	(d)	78	26	106.1	--	97.1	--	2.0
	10	82	28	99.0	80.5	94.7	1.5	1.0
	11	84	29	102.0	85.8	95.8	1.0	1.0
	12	150	66	97.0	75.0	86.0	2.0	1.5
	13	150	66	98.5	72.0	84.9	1.0	1.0
	15	225	107	110.0	73.7	82.3	47.5	15.5
	9	225	107	103.0	72.7	83.5	50.0	17.5
	14	300	149	98.5	74.0	83.6	54.5	15.5
	16 (f)	78	26	107.0	72.5	84.3	5.0	3.5
	17 (f)	150	66	101.0	69.2	78.4	12.0	13.0

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TABLE E-VIII (Cont.)

(a) Longitudinal to final rolling direction.

(b) All specimens unless otherwise marked are from as-received material which was annealed for 1 hour at 2400°F (1316°C).

(c) Strain Rate: 0.005 inch/inch/minute to 0.2% yield strength.

(d) Data reported by vendor.

(e) RA reported was at the location of fracture; the maximum was 37.5%.

(f) Specimen annealed 50 hours at 2600°F (1427°C) at a pressure of $< 1 \times 10^{-5}$ torr.

(g) RA reported was at the location of fracture; the maximum RA was 3.8%.

(h) RA reported was at the location of fracture; the maximum RA was 72.0%.

for 50 hours at 2600°F (1427°C) was considered insignificant. Therefore, no additional heat treatment was selected for the 1.0-inch (2.5-cm)-diameter rod, and it was used in the T-111 Corrosion Loop in the as-received condition.

The room-temperature ductility achieved in the 0.75-inch (1.91-cm)-thick plate after heat-treating for 50 hours at 2600°F (1427°C) was considered acceptable; therefore, this heat treatment was utilized for the 0.75-inch (1.91-cm)-thick, Mo-TZC alloy plate.

Since the machining of the 1.375-inch (3.49-cm)-thick plate and 2.0-inch (5.0-cm)-diameter rod was to be done by electric-discharge-machining techniques, which can be done readily at temperatures on the order of 150°F (66°C), the ductility achieved in both the 1.375-inch (3.49-cm)-thick plate and the 2.0-inch (5.0-cm)-diameter rod at 150°F (66°C) after heat treating 50 hours at 2600°F (1427°C) was considered acceptable. However, even though good ductility was achieved in the 1.375-inch (3.49-cm)-thick plate, it was decided that the non-uniform microstructure observed in the metallographic studies made it undesirable to use this material in the T-111 Corrosion Loop.

The 0.75-inch (1.91-cm)-thick, Mo-TZC alloy plate and the 2.0-inch (5.0-cm)-diameter, Mo-TZC alloy rod were chemically cleaned, wrapped in fresh, Cb-1Zr alloy foil, and subsequently heat treated at 2600°F (1427°C) for 50 hours in a vacuum of $< 1 \times 10^{-5}$ torr.

APPENDIX F

PROCESSING OF Cb-132M ALLOY

The mill products of Cb-132M alloy (Cb-20Ta-15W-5Mo-2Zr-0.13C) required for the program requirements were produced by Universal Cyclops Steel Corporation. A 5 3/8-inch (13.7-cm)-diameter x 10.09-inch (25.63-cm)-long Cb-132M alloy ingot was triple vacuum arc melted from a double EB-melted ingot by Wah Chang Albany and jacketed in a 5 7/8-inch (1.49-cm)-OD, pressed and sintered molybdenum can. The canned ingot was successfully extruded at duPont through a 3 3/4-inch (9.53-cm)-ID die at a temperature of 3120°F (1716°C). The extrusion parameters employed are presented in Table F-I. The extruded bar was conditioned to 3.375-inch (8.57-cm) diameter and sectioned into two lengths. One billet length was stress relieved at 2300°F (1216°C) for one hour in vacuum, machined, recanned in molybdenum, and extruded at 2400°F (1316°C) to approximately 2.25-inch (5.7-cm) diameter. The second billet length was given a recrystallization anneal at 3200°F (1760°C) for one hour in vacuum, machined, recanned in molybdenum, and extruded at 2900°F (1593°C) to approximately 2.25-inch (5.7-cm) diameter. The extrusion parameters for both billets are presented in Table F-II. The latter two extrusions were performed by Nuclear Metals, West Concord, Massachusetts. The extrusion from the second billet was then stress relieved at 2300°F (1260°C) for one hour in vacuum, machined, recanned in molybdenum, and reextruded by Nuclear Metals at 2400°F (1316°C) through a 1.75-inch (4.45-cm)-diameter die. The extrusion parameters are presented in Table F-III.

The 2.25-inch (5.7-cm)-diameter Cb-132M alloy extrusion from billet No. 1 was machined into 2.0-inch (5.08-cm)-diameter rod. The 1.72-inch (4.37-cm)-diameter extrusion from billet No. 2 was decanned, inspected, and machined into 1.0-inch (2.54-cm)-diameter rod.

The chemical analyses of the ingot and stress-relieved rod, as supplied by Universal Cyclops, are shown in Table F-IV. The mechanical properties, also supplied by Universal Cyclops, re presented in Table F-V.

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TABLE F-I

EXTRUSION PARAMETERS FOR THE 5.36-INCH (13.62-cm)-DIAMETER MACHINED Cb-132M ALLOY INGOT[(a)]

Machined Ingot Size	-	5.36-inch (13.62-cm) diameter x 10.16-inch (25.8-cm) long
Ingot Nose Geometry	-	135° included angle
Can Size	-	5.95-inch (15.1-cm)-diameter molybdenum
Leader Block	-	Steel block heated to 2000°F (1093°C)
Follow-up Block	-	2.0-inch (5.1-cm)-thick carbon discs heated to 2000°F (1093°C) followed by one carbon disc at room temperature
Container Size	-	6-inch (15.2-cm) ID
Die Size/Design	-	3.75-inch (9.5-cm) ID/conical
Die Coating	-	ZrO_2
Extrusion Ratio	-	2.56/1
Lubricant	-	Proprietary Glass
Ingot Temperature[(b)]	-	3120°F (1716°C)
Extrusion Pressure	-	76,000 psi (530 MN/m^2)
Extrusion Press Size	-	2.750 tons
Cooling Procedure	-	Air

(a) Extruded at duPont.

(b) Heated by induction in argon.

TABLE F-II

EXTRUSION PARAMETERS FOR THE 3.75-INCH (9.5-cm)-DIAMETER EXTRUDED Cb-132M ALLOY BILLETS[a]

Machined Billets Sizes	-	Billet No. 1[b] - 3.75-inch (9.5-cm) diameter x 12-inch (30.5-cm) long Billet No. 2[c] - 3.75-inch (9.5-cm) diameter x 10-inch (25.4-cm) long
Can Size	-	3.95-inch (9.5-cm)-diameter molybdenum
Leader Block	-	Mild steel heated to 1500°F (816°C)
Container Size	-	4.05-inch (10.3-cm) ID
Die Size/Design	-	2.625-inch (6.67-cm) ID/conical
Die Coating	-	ZrO_2
Extrusion Ratio	-	2.4/1
Lubricant	-	Glass
Billet Temperature[d]	-	Billet No. 1 - 2400°F (1316°C) Billet No. 2 - 2900°F (1593°C)
Extrusion Pressure	-	Billet No. 1 - 750-690 tons runout Billet No. 2 - 510-370 tons runout
Extrusion Press Size	-	1400 tons
Cooling Procedure	-	Air

(a) Extruded at Nuclear Metals, West Concord, Mass.

(b) Billet No. 1 was used to make the 2-inch (5.1-cm)-diameter bar.

(c) Billet No. 2 was used to make the 1-inch (2.54-cm)-diameter bar.

(d) Heated by induction in argon.

TABLE F-III

EXTRUSION PARAMETERS FOR THE 2.0-INCH (5.1-cm)-DIAMETER EXTRUDED Cb-132M ALLOY BILLET[(a)]

Machined Billet Size	-	Billet No. 2 - 2.0-inch (5.1-cm) diameter x 12-inch (30.5-cm) long
Can Size	-	3.95-inch (9.5-cm)-diameter molybdenum
Leader Block	-	None
Follow-up Block	-	Mild steel heated to 900°F (482°C)
Container Size	-	4.05-inch (10.3-cm) ID
Die Size/Design	-	1.75-inch (4.4-cm) ID/conical
Die Coating	-	ZrO_2
Extrusion Ratio	-	3.0/1
Lubricant	-	Glass
Billet Temperature[(b)]	-	2400°F (1316°C)
Extrusion Pressure	-	700 tons
Extrusion Press Size	-	1400 tons
Cooling Procedure	-	Air

(a) Extruded at Nuclear Metals, West Concord, Mass.

(b) Induction-heated in argon.

TABLE F-IV

CHEMICAL ANALYSES OF Cb-132M ALLOY (HEAT NO. 66-95119)
PRODUCED BY UNIVERSAL CYCLOPS STEEL CORPORATION

	Material					
	Ingot				2-Inch(5.1-cm)-Dia. Rod	
	Top-Center	Top-Mid-radius	Top-Edge	Bottom-Center	Center	Edge
%						
C	0.13	0.14	0.15	0.13	0.124	0.119
Ta	19.82	19.53	19.44	20.36	--	--
W	14.9	13.9	14.0	15.3	--	--
Mo	5.01	5.06	5.02	4.72	--	--
Zr	1.97	2.13	2.16	1.75	--	--
ppm						
O	70	<50	<50	<50	28	35
N	30	30	35	40	76	82
H	2.8	2.1	1.9	2.5	4.0	3.1
Al	<20	<20	<20	<20	--	--
B	<1	<1	<1	<1	--	--
Cd	<5	<5	<5	<5	--	--
Co	<10	<10	<10	<10	--	--
Cr	<20	<20	<20	<20	--	--
Cu	<40	<40	<40	<40	--	--
Fe	<50	50	50	50	--	--
Mg	<20	<20	<20	<20	--	--
Mn	<20	<20	<20	<20	--	--
Ni	<20	<20	<20	<20	--	--
Pb	<20	20	<20	<20	--	--
Si	<50	<50	<50	<50	--	--
Sn	30	100	25	35	--	--
Ti	<40	<40	<40	<40	--	--
V	<20	<20	<20	<20	--	--

TABLE F-V

MECHANICAL PROPERTIES[a] OF Cb-132M ALLOY (HEAT NO. 66-95119)
PRODUCED BY UNIVERSAL CYCLOPS STEEL CORPORATION

Specimen Location	Room Temperature Tensile Properties[b]: Ultimate Strength 1,000 psi	0.2% Yield Strength 1,000 psi	Reduction in Area %	Elong. %	Stress-Rupture Life at 2200°F (1204°C) and 30,000 psi in Vacuum: Life Hours	Reduction in Area %	Elong. %	Vacuum at Test Temp. Torr
1-Inch (2.54-cm)-Dia. Rod[c]	139.0	115.0	14	11	10.4	43	32	2.9×10^{-6}
	138.5	116.5	13	11	10.1	42	19	1.7×10^{-6}
2-Inch (5.1-cm)-Dia. Rod	130.0[c,d]	116.5	3.0	2	78.4[e]	40	25	7×10^{-7}
	132.0[c,d]	116.4	1.5	2	38.1[f,c]	42	15	9×10^{-6}

(a) Tensile and stress-rupture specimens were machined from Cb-132M alloy rod following annealing for one hour at 2400°F (1316°C). The axes of the specimens were parallel to the rolling direction.

(b) Tensile properties were determined using a strain rate of 0.005 inch/inch/min up to 0.2% offset and then 0.050 inch/min head rate to fracture.

(c) The specimens had a 0.160-inch (0.41-cm) diameter with a 0.80-inch (2.04-cm)-gauge length.

(d) Specimen failed in the radius.

(e) Total hours, specimen adapter failed after 37.9 hours and test was restarted after replacement of adapter. The specimen had a 0.250-inch (0.635-cm) diameter with a 1.45-inch (3.68-cm)-gauge length.

(f) Total hours, threads failed on specimen after 11.6 hours and test was restarted after modifying specimen to buttonhead design.

Because of the marginal room temperature ductility of the 2-inch (5.08-cm)-diameter Cb-132M alloy rod, a heat-treatment study was conducted to establish final annealing conditions that would result in an optimum combination of low-temperature ductility and elevated-temperature strength.

A heat treatment of one hour at 2600°F (1427°C) was selected for the 2-inch (5.08-cm)-diameter Cb-132M alloy rod. This study showed that this heat treatment results in improved room-temperature ductility without appreciable recrystallization or reduction in the yield strength of the Cb-132M alloy.

The Cb-132M alloy as supplied by Universal Cyclops had been heat treated for one hour at 2400°F (1316°C) and had a room-temperature ductility somewhat less than desirable for machining and utilization in the Corrosion Loop. It was anticipated that a slightly higher-temperature heat treatment would result in increased carbide precipitation and reduce the amount of carbon in solid solution, which would improve the room-temperature ductility without an appreciable loss in yield strength. Approximately 0.25-inch (0.64-cm)-thick slices were cut from the end of the Cb-132M alloy rod and subsequently sectioned into pie-shaped specimens. After chemical cleaning, the specimens were heat-treated for one hour at 2500°, 2600°, 2700°, 2800°, and 2900°F (1371°, 1427°, 1482°, 1538°, and 1593°C) at a pressure of less than 1×10^{-5} torr (1.3×10^{-3} N/m^2) and then quenched in helium.

The microstructures and microhardnesses of the Cb-132M alloy before and after the one-hour heat-treatments are shown in Figure F-1. The first signs of slight recrystallization in the severely worked regions of the bar were observed in the specimen heat treated for one hour at 2700°F (1482°C). Significant agglomeration of the carbide precipitate was observed after a heat treatment of one hour at 2900°F (1593°C), Figures F-2 through F-4. The microhardness data indicate a rapid decrease in hardness after a one-hour heat treatment at 2700°F (1482°C). From the microhardness and metallographic data, a temperature of less than 2700°F (1482°C) was suggested for a one-hour heat treatment.

The effects of longer-time, lower-temperature heat treatments were

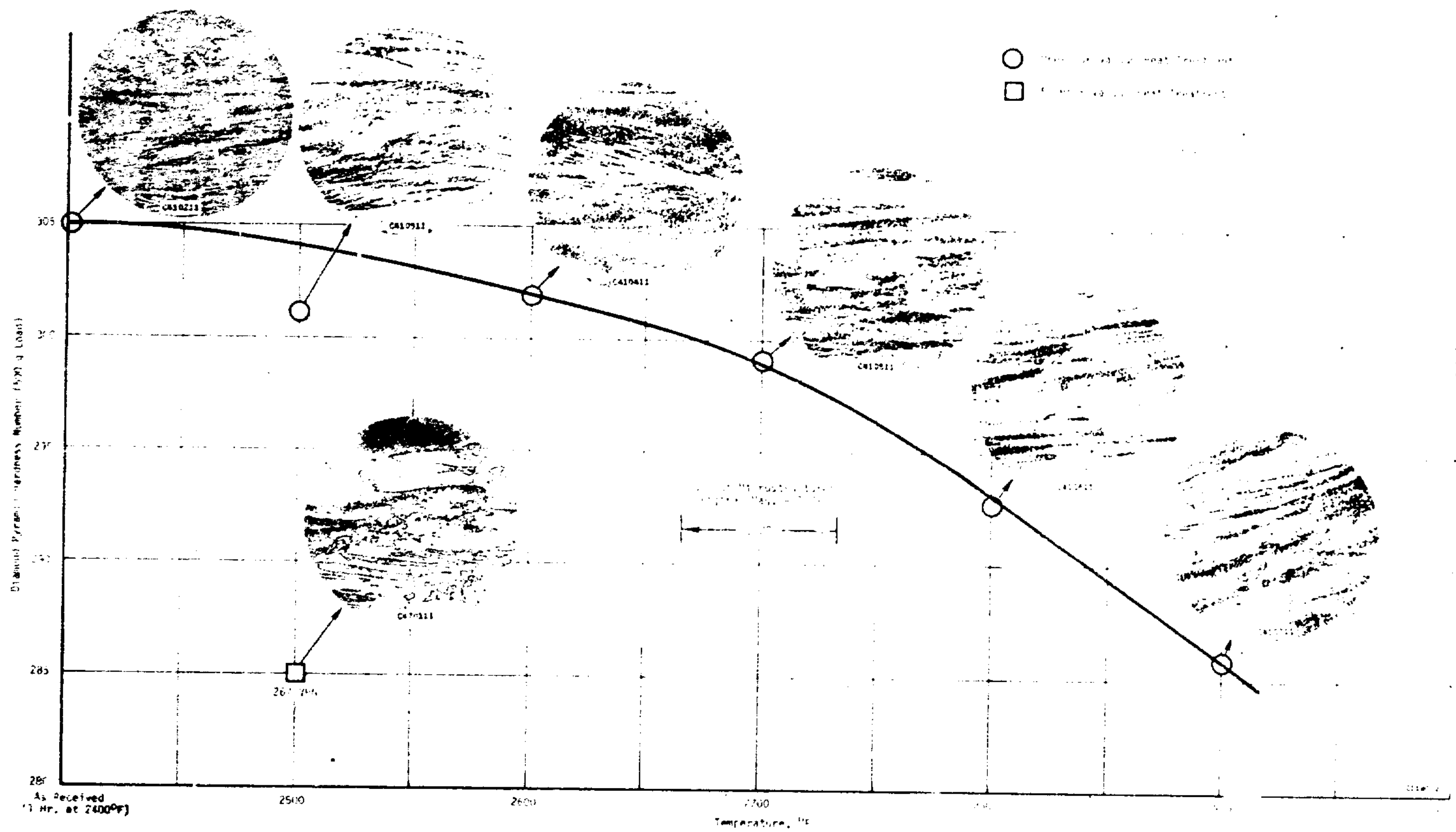
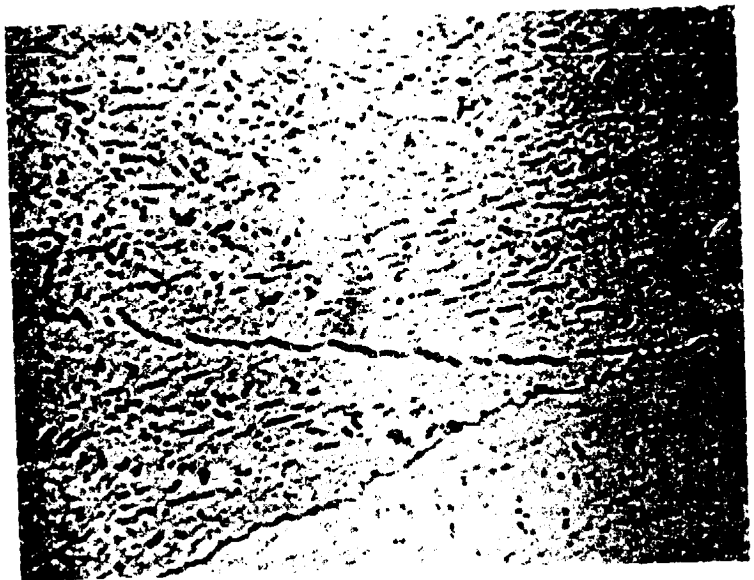
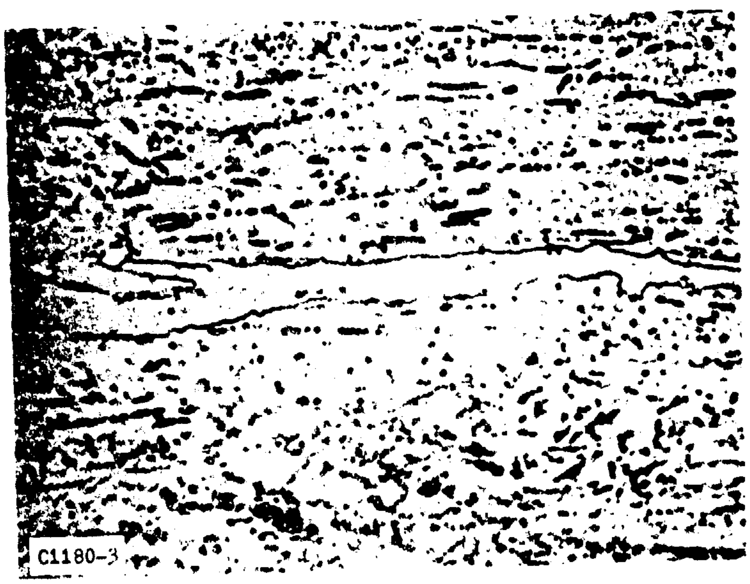


Figure F-1. Microhardness and Microstructure of Longitudinal Specimens Cut From 2.0-Inch (5.1-cm)-Diameter Cb-132M Alloy Rod.
Etchant: 20mlHNO_3-20mlHF-60mlGlycerine



a) As-Received (Annealed 1 Hour at 2400°F [1316°C])



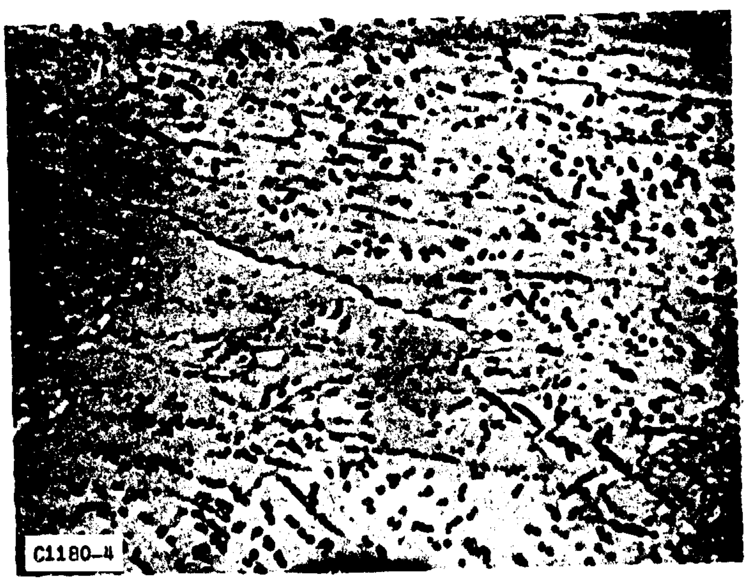
b) Annealed 1 Hour at 2500°F (1371°C)

Figure F-2. Longitudinal Microstructure of 2.0-Inch (5.1-cm)-Diameter Cb-132M Alloy Rod.

Etchant: 60mlGlycerine-20mlHNO$_3$-20mlHF

Mag.: 2000X a) C410212, b) C410313

a) Annealed 1 Hour at 2600°F (1427°C)

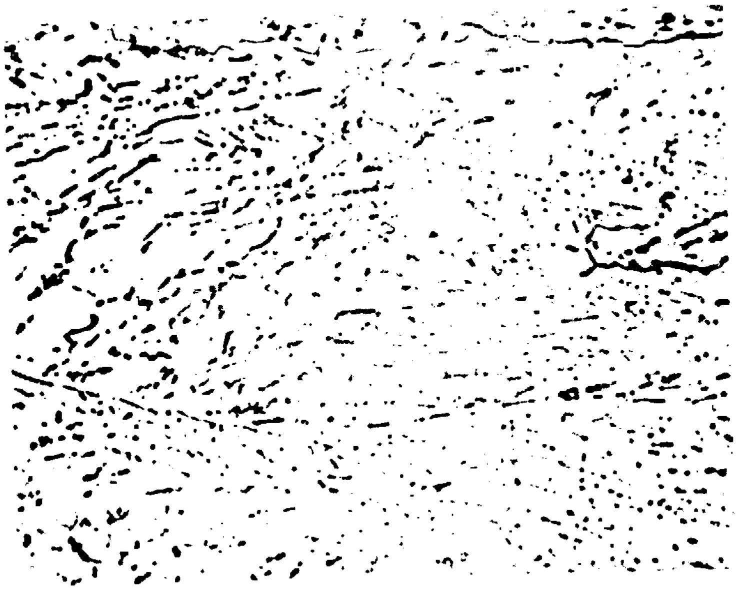


b) Annealed 1 Hour at 2700°F (1482°C)

Figure F-3. Longitudinal Microstructure of 2.0-Inch (5.1-cm)-Diameter Cb-132M Alloy Rod.

Etchant: 60ml Glycerine-20ml HNO_3-20ml HF

Mag.: 2000 X a) C410413, b) C410512



a) Annealed 1 Hour at 2800°F (1538°C)

b) Annealed 1 Hour at 2900°F (1593°C)

Figure F-4. Longitudinal Microstructures of 2.0-Inch (5.1-cm)-Diameter Cb-132M Alloy Rod.

Etchant: 60mlGlycerine 20ml HNO_3 20mlHF

Mag.: 2000X a) C410613, b) C410712

evaluated by heat treating a sample for 50 hours at 2500°F (1371°C). The microstructure, shown in Figures F-1 and F-5, indicates significant recrystallization and agglomeration of the carbide precipitate. These changes are reflected in the low microhardness given in Figure F-1.

The final heat-treatment selection was determined by evaluating the tensile behavior of specimens heat treated for one hour at 2500°F (1371°C) and one hour at 2600°F (1427°C). Buttonhead tensile specimens were machined from the 2-inch (5.1-cm)-diameter Cb-132M rod such that the longitudinal axes of the rod and specimens were parallel. The finished tensile specimens were chemically cleaned and carefully inspected for surface defects using fluorescent-penetrant techniques. Three tensile specimens were vacuum heat treated at pressures less than 1×10^{-5} torr (1.3×10^{-3} N/m^2) for one hour at 2500°F (1371°C), three for one hour at 2600°F (1427°C), and the remaining specimens were left in the as-received condition (one hour at 2400°F (1316°C). The tensile tests were conducted in air at room temperature to 225°F (107°C) and were performed in accordance with ASTM Designation E8-57T, "Methods of Tension Testing of Metallic Materials." A strain rate of 0.005 inch/inch/minute up to 0.6 percent offset and then 0.050 inch/inch/minute to fracture was used; the yield strength was determined by the 0.02 percent and 0.2 percent offset methods. The results of these tests are shown in Table F-VI along with the tensile data supplied by Universal Cyclops Steel Corporation.

Reasonable ductility was observed in the as-received Cb-132M alloy material which was annealed one hour at 2400°F (1316°C) and tensile tested at a temperature of 225°F (107°C). Both thermal heat treatments, one hour at 2500°F (1371°C) and one hour at 2600°F (1427°C), resulted in good room-temperature ductility, and the specimens which were heat treated at 2600°F (1427°C) exhibited slightly higher ductility than those heat treated at 2500°F (1371°C). In all cases, no significant reductions in yield strength were observed. From these results and the earlier microstructure and microhardness results, a thermal heat treatment of one hour at 2600°F (1427°C) was selected for the 2-inch (5.08-cm)-diameter Cb-132M alloy rod. Subsequently,

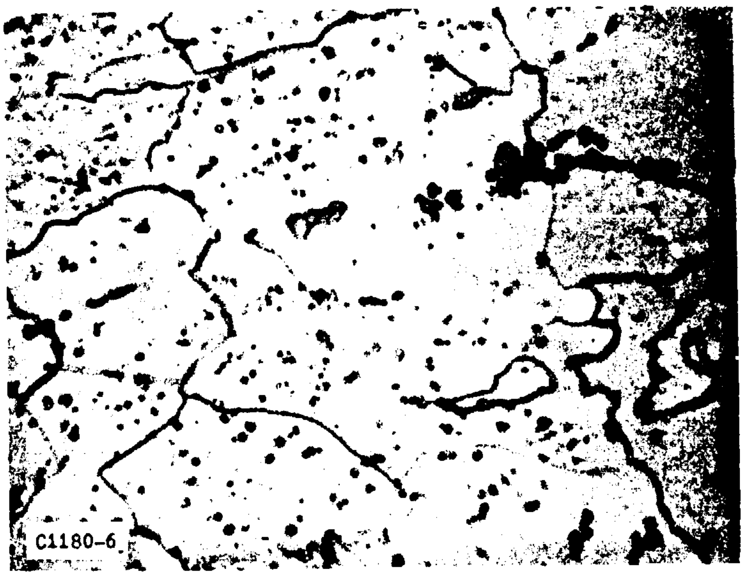


Figure F-5. Longitudinal Microstructure of 2.0-Inch (5.1-cm)-Diameter Cb-132M Alloy Rod Annealed 50 Hours at 2500°F (1371°C).

Etchant: 60ml Glycerine-20ml HNO_3-20ml HF

Mag.: 2000X (C470112)

TABLE F-VI

TENSILE PROPERTIES OF 2-INCH (5.1-cm)-DIAMETER Cb-132M ALLOY ROD[a]

Specimen Number	Test Temperature °F	Test Temperature °C	Ultimate Tensile Strength psi x 10^{-3}	0.02% Yield Strength[b] psi x 10^{-3}	0.2% Yield Strength[b] psi x 10^{-3}	Reduction in Area (%)	Elongation % in One Inch (2.54 cm)
AS-RECEIVED CONDITION (1 HOUR AT 2400°F [1316°C])							
1[c]	78	26	130.0	Not Reported	116.5	3.0	2.0
2[c]	78	26	132.0	Not Reported	116.4	1.5	2.0
3[d]	150	66	123.0	106.0	112.0	4.0	3.0
4[d]	150	66	132.0	109.0	114.0	4.7	3.0
5	225	107	126.0	101.0	107.0	36.0	15.0
HEAT-TREATED ONE HOUR AT 2500°F [1371°]							
6	150	66	127.0	102.0	109.0	33.0	14.0
7	78	26	132.0	108.0	115.0	15.0	11.0
8	78	26	131.0	110.0	114.0	27.0	14.0
HEAT-TREATED ONE HOUR AT 2600°F [1427°C]							
9	150	66	123.0	106.0	106.0	44.0	17.0
10	78	26	132.0	109.0	114.0	44.0	17.0
11	78	26	132.0	108.0	114.0	20.0[e]	13.0

(a) Longitudinal direction.

(b) Strain Rate: 0.005 inch/inch/minute up to 0.6% offset and then 0.050 inch/inch/minute to fracture.

(c) Data reported by Universal Cyclops Steel Corp.

(d) Failed at radius - minimum reduction area at center.

(e) Not at location of fracture.

the 2-inch (5.08-cm) diameter Cb-132M alloy rod was wrapped in new Cb-1Zr alloy foil and vacuum annealed for one hour at 2600°F (1427°C).

The 1-inch (2.54-cm)-diameter rod had satisfactory room-temperature ductility in the as-received condition (2400°F [1316°C]/1 hour).

APPENDIX G

T-111 BELLOWS FABRICATION

As indicated in Section IV, Fabrication, of this report, T-111 bellows were fabricated for use in the construction of the metering and isolation valves for the T-111 Corrosion Test Loop. The T-111 bellows were of good quality, however, the decision was made to take the more conservative and demonstrated approach of using Cb-1Zr bellows from the same lot used in the valves of the successful 5000-hour Cb-1Zr Corrosion Loop Test. The higher-strength T-111 alloy bellows were not required because of the low operating temperature and low stress levels in the convolution walls. Even through the T-111 bellows were not used, a brief description of the fabrication of these bellows is included below since these were the first bellows made from this high-strength alloy. The information gained during the fabrication of these 0.52-inch (1.3-cm)-OD bellows was extremely useful in the subsequent fabrication of 0.85-inch (2.2-cm)-OD T-111 bellows for the larger refractory metal valves which were evaluated in another recent loop test.[31]

The T-111 bellows for the T-111 Corrosion Test were formed from tube blanks of the following dimensions, 0.375-inch (0.95-cm) OD x 0.008-inch (0.02-cm) wall thickness x 6.75-inch (17.1-cm) length. Because of the thin walls of the bellows' blanks, the interstitial element concentrations in this material were substantially higher than the tubing and pipe used in the construction of the T-111 loop proper. Following the final recrystallization heat treatment of 1 hour at 2700°F (1482°C) the average analysis of the T-111 blanks was as follows: 203 ppm oxygen, [illegible]07 ppm carbon, 36 ppm nitrogen, and 3 ppm hydrogen. The relatively high oxygen concentration and the attendant risk of attack by lithium was another reason for selecting Cb-1Zr bellows for use in the fabrication of the T-111 Corrosion Loop valves.

The T-111 bellows were hydraulically formed with an internal pressure of approximately 2400 psi (16.5×10^6 N/m^2) by Miniflex Corporation, Van Nuys,

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(31) Harrison, R. W. and Holowach, J., Refractory Metal Valves for 1900°F Service in Alkali Metal Systems, NASA CR-1810, 1970.

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California. Various stages of the formation of the convolutions are illustrated in Figure G-1. A close-up view of the bellows is shown in Figure G-2, which illustrates the uniformity of the convolutions and the smooth texture of the surface. The metallographic appearance and the uniformity of the wall thickness of the bellows' convolutions are illustrated in Figure G-3. The maximum variation in wall thickness between the apex and the side wall of the convolutions was less than 0.001 inch (0.002 cm). The grain size in the convolutions corresponded to ASTM 7-8. Microhardness measurements were made on the cross section of the tube blanks and on the formed bellows. Diamond Pyramid Hardness values of 250 were obtained on the tube blanks and values ranging from 325 to 350 were obtained on the bellows' convolutions.

The axial deflections of two T-111 bellows and a Cb-1Zr bellows as a function of load were determined and are presented in Table G-I. No permanent set was observed in these room temperature tests of the bellows in the 0.10-inch (0.25-cm) travel necessary for valve operation.

In summary, examination of the T-111 bellows indicated that they were of excellent quality with the sole negative factor being the relatively high oxygen and carbon concentrations of this material.

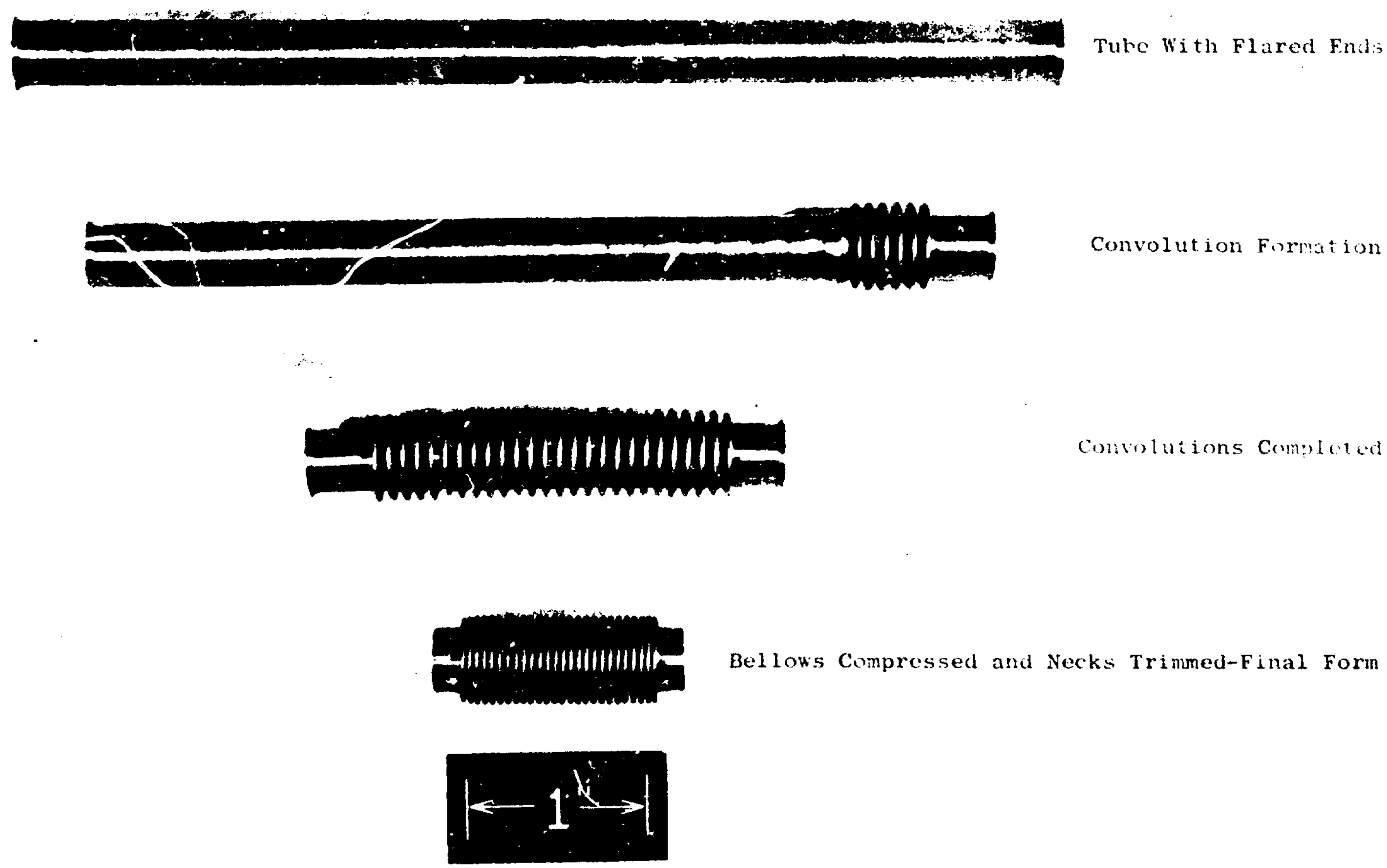


Figure G-1. Stages in the Formation of T-111 Alloy Bellows. (Orig. Neg. C65082016)

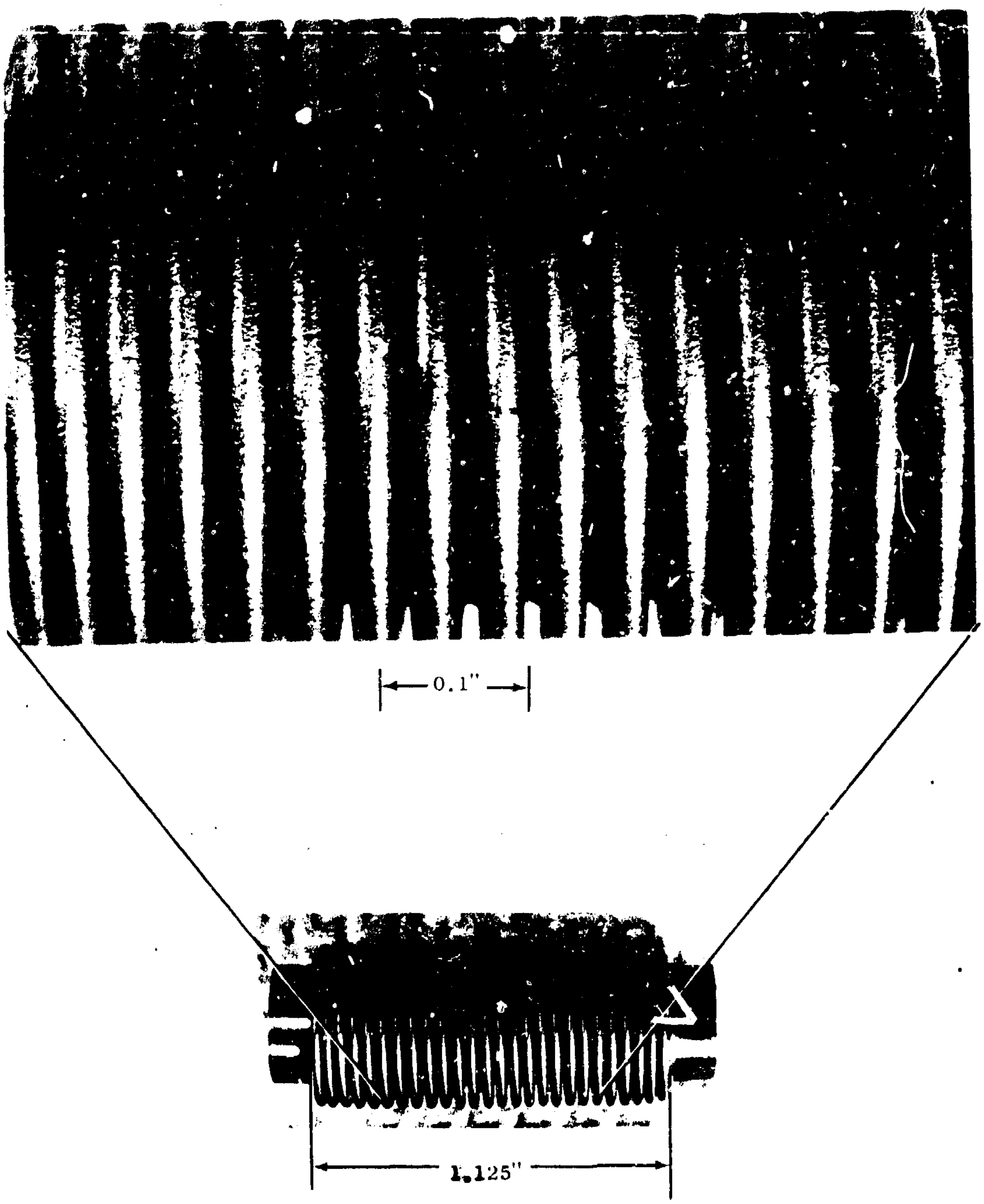


Figure G-2. T-111 Alloy Valve Bellows. (Top - C65082015)
(Bottom - C65082006)

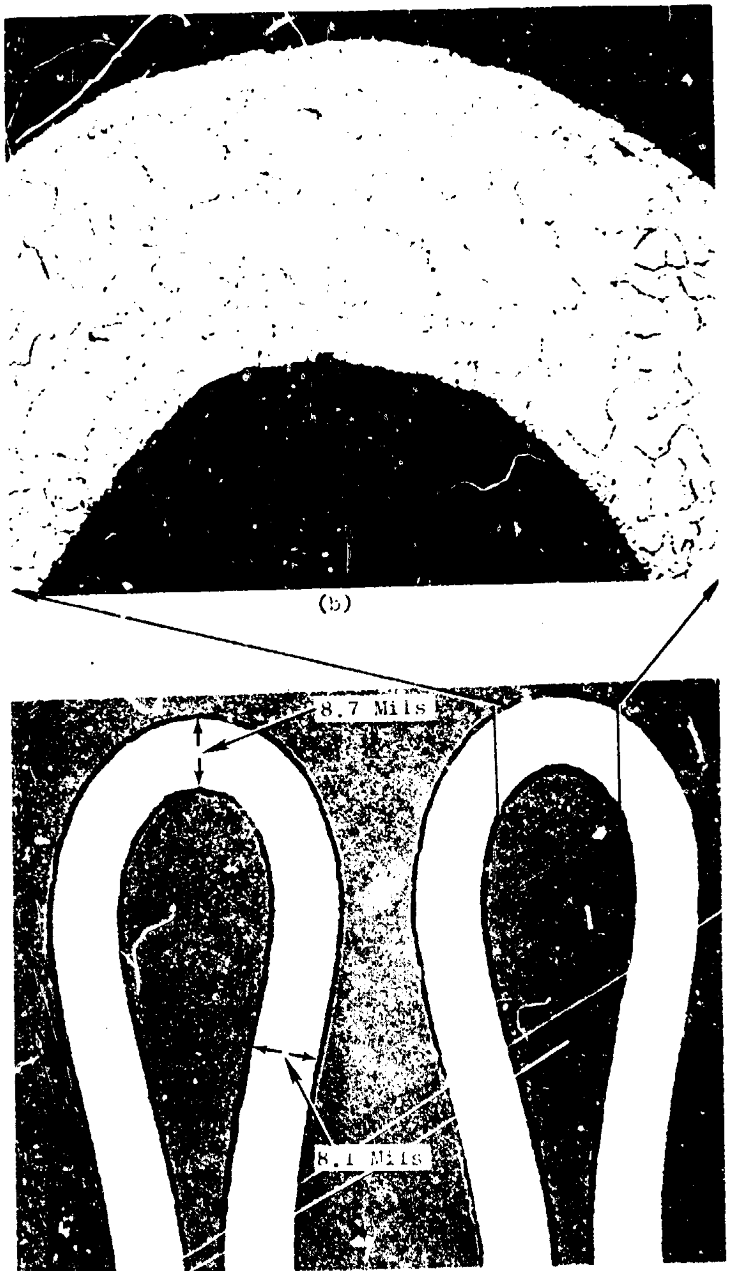


Figure G-3. (a) A Transverse Section of T-111 Alloy Bellows' Convolutions Showing the Uniformity of the Wall Thickness (Orig. A830213).
(b) A Transverse Section at the Apex of a Convolution Indicating an ASTM Grain Size 7-8 (Orig. A830212).

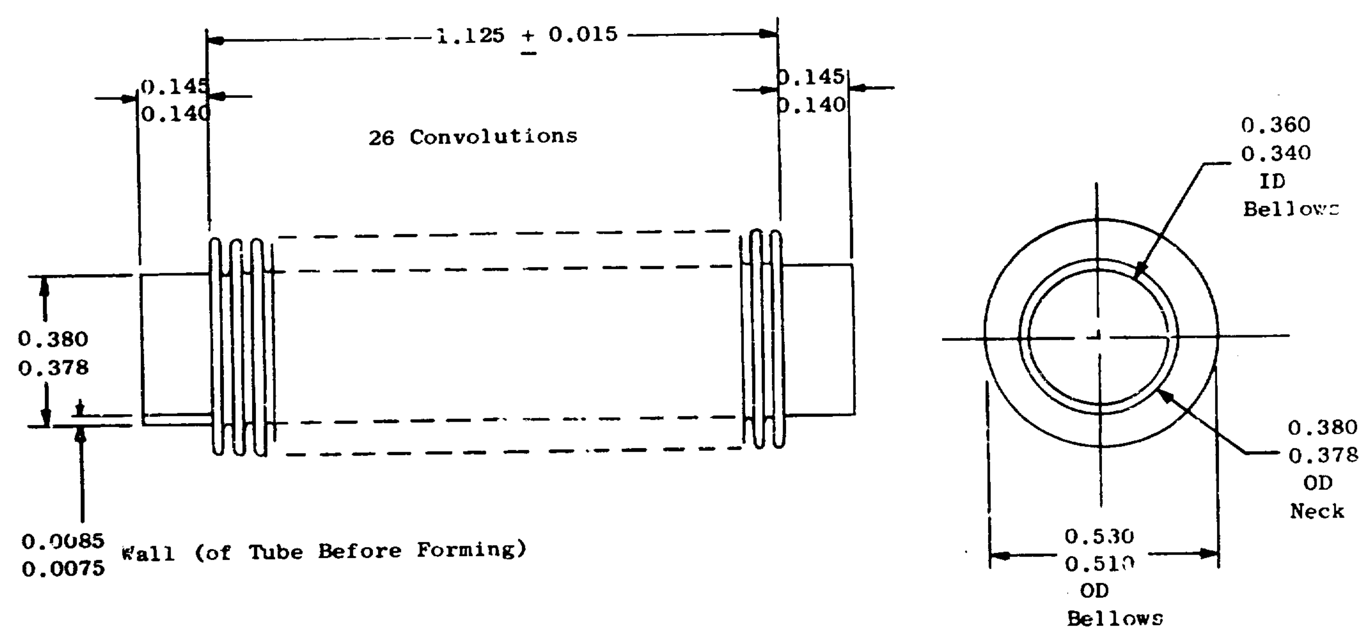
Etchant: $40\% HNO_3$-$40\% HF$-$20\% HCl$ Orig. Mag.: (a) 50X
(b) ∠50X

TABLE G-I

ROOM TEMPERATURE COMPRESSIVE PROPERTIES OF T-111 ALLOY AND Cb-1Zr ALLOY VALVE BELLOWS

Alloy Specimen	Elastic Spring Rate Lbs/Inch	Total Deflection at Yield Inch	Load at Yield Lbs	Permanent Set In/Full 0.1 Inch Deflection*	Load Required for Full 0.1 Inch Deflection* Lbs
T-111-1	428	0.140	60	0	42.8
T-111-2	443	0.131	58	0	44.3
Cb-1Zr-1	185	0.112	20.8	0	18.5

* 0.1-Inch Deflection Normal Travel Range of Valve.



APPENDIX H

CALIBRATION OF THE PARTIAL PRESSURE GAS ANALYZER

The method used for calibration of the mass spectrometer and subsequent calculations of partial pressures involves the use of both the ionization gauge and the mass spectrometer in a procedure such that the total pressure is determined by the ionization gauge reading, corrected for relative concentrations of various gas species, with the relative concentrations obtained from the mass spectrum. The reason for this approach is that the mass spectrometer cannot be relied upon to give accurate absolute partial pressure values over a long time period. This is due mainly to instabilities in the electron multiplier. The ionization gauge, on the other hand, will give only approximate values of the total pressure unless relative partial pressures are known. Details of the calibration and data analysis procedures are given elsewhere.[32]

Calibration of the mass spectrometer was made before installation of the loop in the chamber. The tube was mounted on the chamber, and the empty chamber was evacuated without the spool piece. After chamber had been baked out and prior to calibration of the analyzer, a base pressure of 3.9×10^{-9} torr (5.2×10^{-7} N/m^2) was obtained.

The analyzer was calibrated for hydrogen, helium, nitrogen, and argon by admitting the pure gas to the chamber through the variable leak valve, which was shown previously in Section VI.B. When the pressure had stabilized, a reading of the ionization gauge was made, and a mass spectrum was obtained. This process was repeated at various pressures for each of the four calibrating gases. From these data, the sensitivity of the mass spectrometer for each of the four gases was calculated. The mass spectrometer sensitivity, $S_{ms}(x)$ for any pure gas (x), is defined as

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(32) Lyon, T. F., Effects of Vacuum Level on Solar Receiver Materials, Final Report on Contract NAS 3-11828, October 10, 1970.

$$S_{ms}(x) = \frac{I_{ms}(x)}{i_{ms} P(x)}$$

where $I_{ms}(x)$ is the positive ion current of the mass spectrometer for a particular peak (usually the parent peak) in the pure gas mass spectrum, i_{ms} is the electron emission current, and $P(x)$ is the gas pressure.

The mass spectrometer sensitivity relative to nitrogen is given in Table H-I for each gas normally found in the residual gas mass spectrum. Values for gases other than those for which direct calibration was made were obtained by extrapolation or interpolation. Also shown in Table H-I are the ionization gauge sensitivities relative to nitrogen. These values were obtained from various literature sources.

TABLE H-I

RELATIVE SENSITIVITY OF MASS SPECTROMETER AND IONIZATION GAUGE

Parent Specie	m/e	Mass Spectrometer Sensitivity Relative to N_2	Ionization Gauge Sensitivity Relative to N_2
H_2	2	1.61	0.42
He	4	0.13	0.19
CH_4	16	1.67	1.07
H_2O	18	1.29	0.89
CO or N_2	28	1.00	1.00
O_2	32	0.72	0.85
Ar	40	0.97	1.56
CO_2	44	0.74	1.37

This process was repeated at various pressures for each of the four calibrating gases. From these data, the sensitivity of the mass spectrometer for each of the four gases was calculated. The mass spectrometer sensitivity, $S_{ms}(x)$ for any pure gas (x), is defined as

$$S_{ms}(x) = \frac{I_{ms}(x)}{i_{ms} P(x)}$$

where $I_{ms}(x)$ is the positive ion current of the mass spectrometer for a particular peak (usually the parent peak) in the pure gas mass spectrum, i_{ms} is the electron emission current, and P(x) is the gas pressure.

The mass spectrometer sensitivity relative to nitrogen is given in Table H-I for each gas normally found in the residual gas mass spectrum. Values for gases other than those for which direct calibration was made were obtained by extrapolation or interpolation. Also shown in Table H-I are the ionization gauge sensitivities relative to nitrogen. These values were obtained from various literature sources.

APPENDIX I

CALIBRATION OF W-3Re/W-25Re THERMOCOUPLE WIRE

Calibration of the thermocouple wire used on the T-111 Corrosion Loop was conducted in a high-vacuum environment with sample thermocouples made from the same spools of wire which were used to instrument the loop. This is the same wire used to instrument the Cb-1Zr Corrosion Loop. A complete description of the apparatus used, procedures, and test results have been previously reported.[33]

The W-3Re (Material Control No. 465) was purchased from the Lamp Metals Components Department of the General Electric Company, Cleveland, Ohio. The W-25Re wire (Material Control No. 453) was purchased from Hoskins Manufacturing Company, Detroit, Michigan.

Calibration of the thermocouple wire was performed in the ultrahigh-vacuum thermocouple test facility shown in Figure I-1. Two Pt/Pt-10Rh reference thermocouples were used for the calibration. Four sample thermocouples of W-3Re vs W-25Re were calibrated against the platinum couples in the thermocouple bundle shown in Figure I-2. Three of the hot junctions were formed by spot-welding the individual wires to the Cb-1Zr tube in the same manner as was used in the loop instrumentation. The fourth thermocouple was formed by twisting the alloy wires together. Both types of junctions are evident in Figure I-2.

Calibration data were obtained during three temperature cycles. During these runs, the pressure in the calibration chamber varied between a low of 2×10^{-8} torr (3×10^{-6} N/m^2) at 400°F (204°C) and a high of 6×10^{-6} torr (8×10^{-4} N/m^2) at 2400°F (1320°C).

In the reduction of the calibration data, a difference parameter is used in order to facilitate curve plotting and to permit use of an expanded

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(33) Hoffman, E. E. and Holowach, J, "Cb-1Zr Rankine System Corrosion Test Loop," Potassium Corrosion Test Loop Development, NASA CR-1509, May 1968.

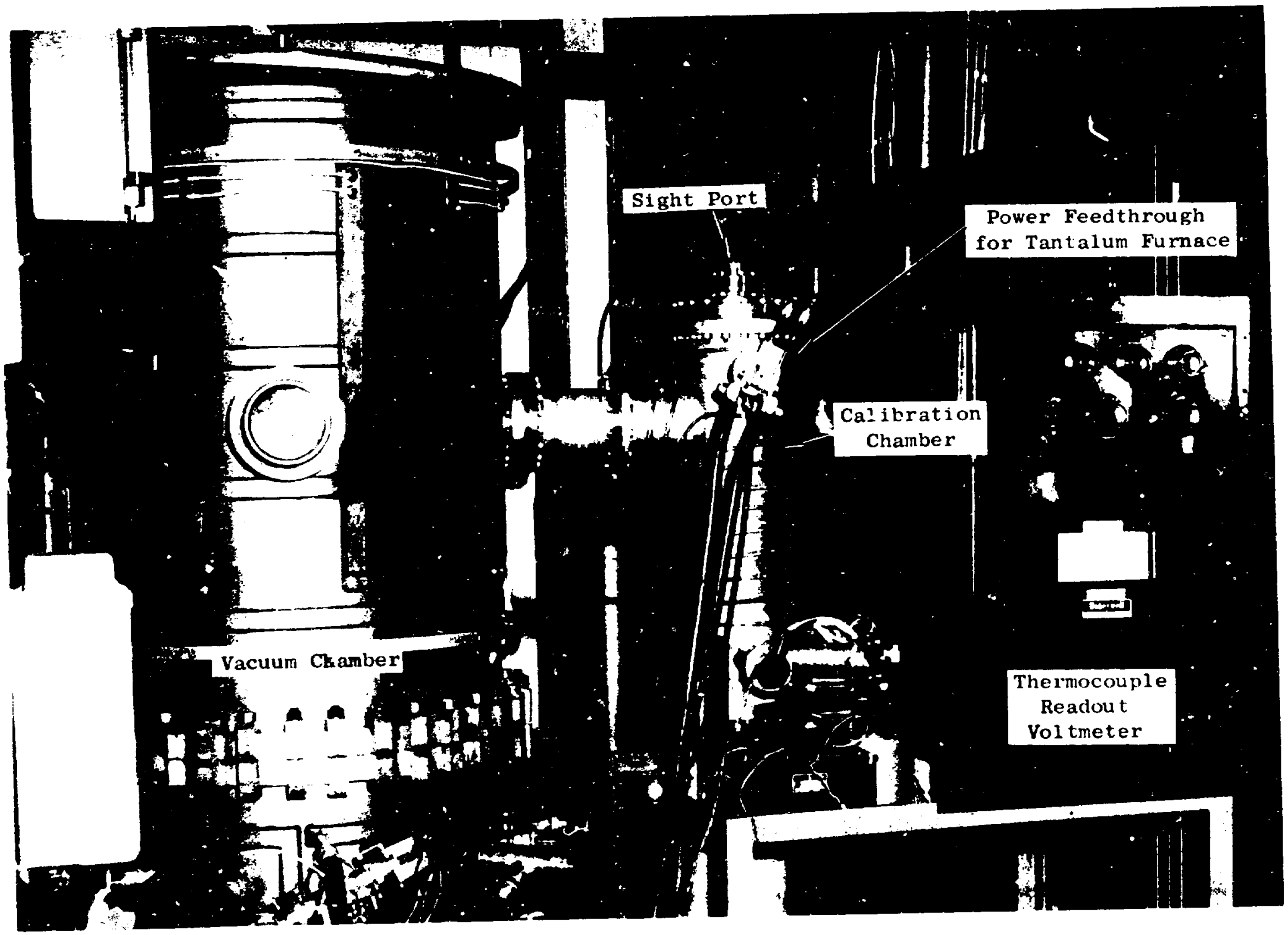

Figure I-1. Thermocouple Calibration Furnace Attached to the 24-Inch (61-cm)-Diameter Getter-Ion Pumped Vacuum Chamber. (Orig. C65040528)

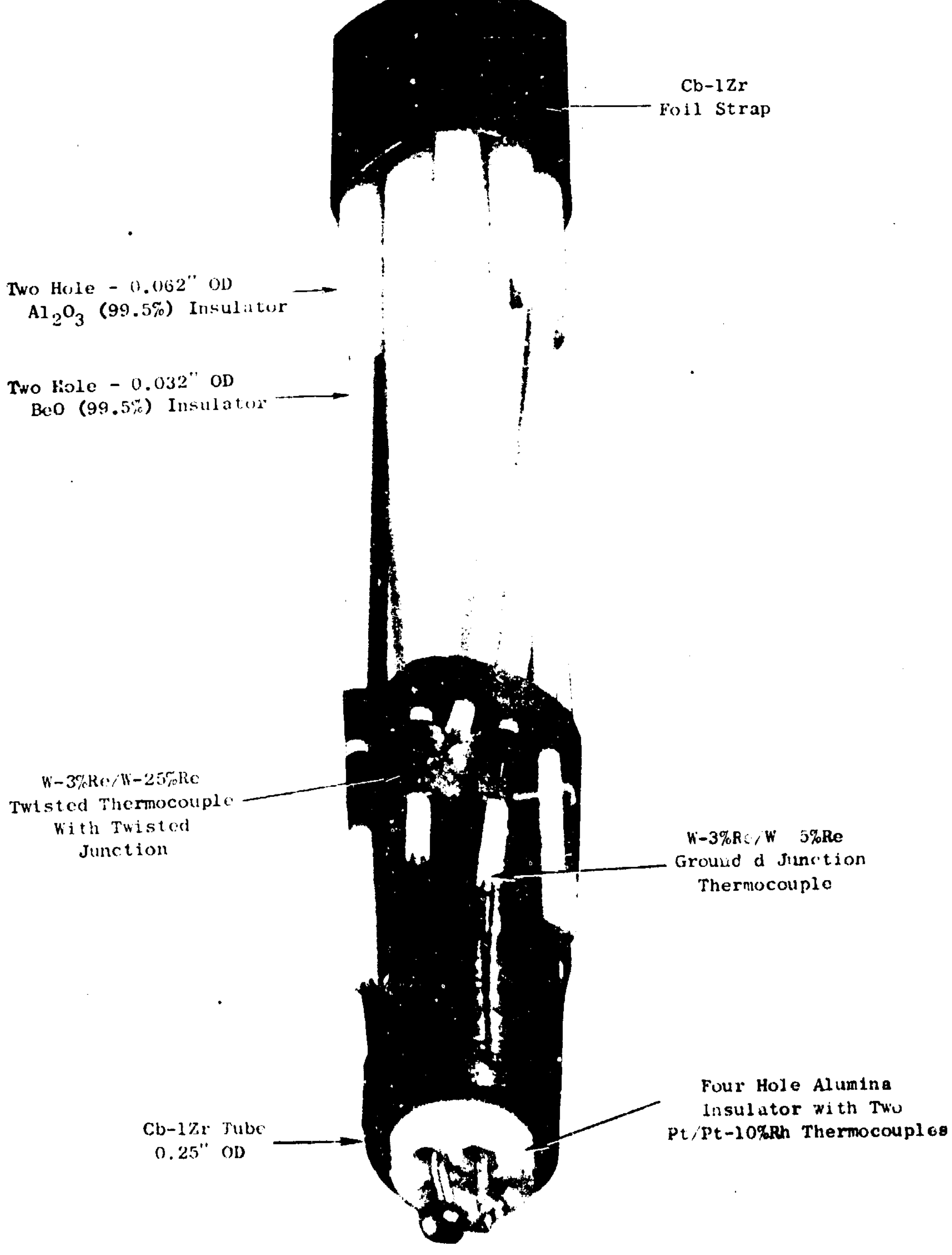

Figure 1-2. Thermocouple Bundle Used in Calibration of W-3Re/W-25Re Thermocouple Wire. (Orig. C65031521)

scale. The difference parameter (in millivolts) is defined as

$$\text{difference parameter} = \frac{T(^{\circ}F)}{100} - \text{EMF (mv)}$$

The curve shown in Figure I-3 is an average of all data points taken during three temperature cycles over the range from 80° to 2400°F (27° to 1320°C) for two thermocouples with different hot junction configurations but from the same spools of alloy wire. All but 8 of 35 stable points fall within 4°F of this line. Maximum deviation of any stable point from the line (up to 2200°F, 1200°C) is 9°F (5°C). The actual values used to define the curve are listed in Table I-I. These are not actual test points but were picked off of the best line through the test data so that linear interpolation between any two points would cause a maximum error of 0.5°F (0.28°C).

It is of interest to compare the thermocouple output, as obtained in this calibration, with similar data from other sources. A table of EMF vs temperature for W-3Re/W-25Re thermocouples is available from Engelhard Industries, Inc.[34] A comparison of the thermocouple EMF from the present calibration with the Engelhard data is given in Table I-II. It should be noted that the general variation of EMF with temperature is the same for both sets of data. The difference between the present calibration and the Engelhard data reaches a maximum positive value at about 600°F (320°C) with a difference of 0.100 mv which is equivalent to a temperature difference of about 10°F (5.6°C). At about 900°F (480°C) the EMF's are the same, and at higher temperatures, the Engelhard data shows higher EMF than the present calibration. The difference reaches the largest negative value at about 2000°F (1100°C) with a difference of -0.152 mv which is equivalent to a temperature difference of about 14.8°F (8.2°C).

It should be emphasized that this comparison does not confirm the accuracy of the present calibration. It does serve to exemplify the agreement which might be expected between W-3Re/W-25Re thermocouples of different lots and subjected to different processing history.

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(34) Electromotive Force vs Temperature for W-3Re/W-25Re Thermocouples, Engelhard Industries, Inc., Research and Development Division - Final Evaluation of November 1, 1965.

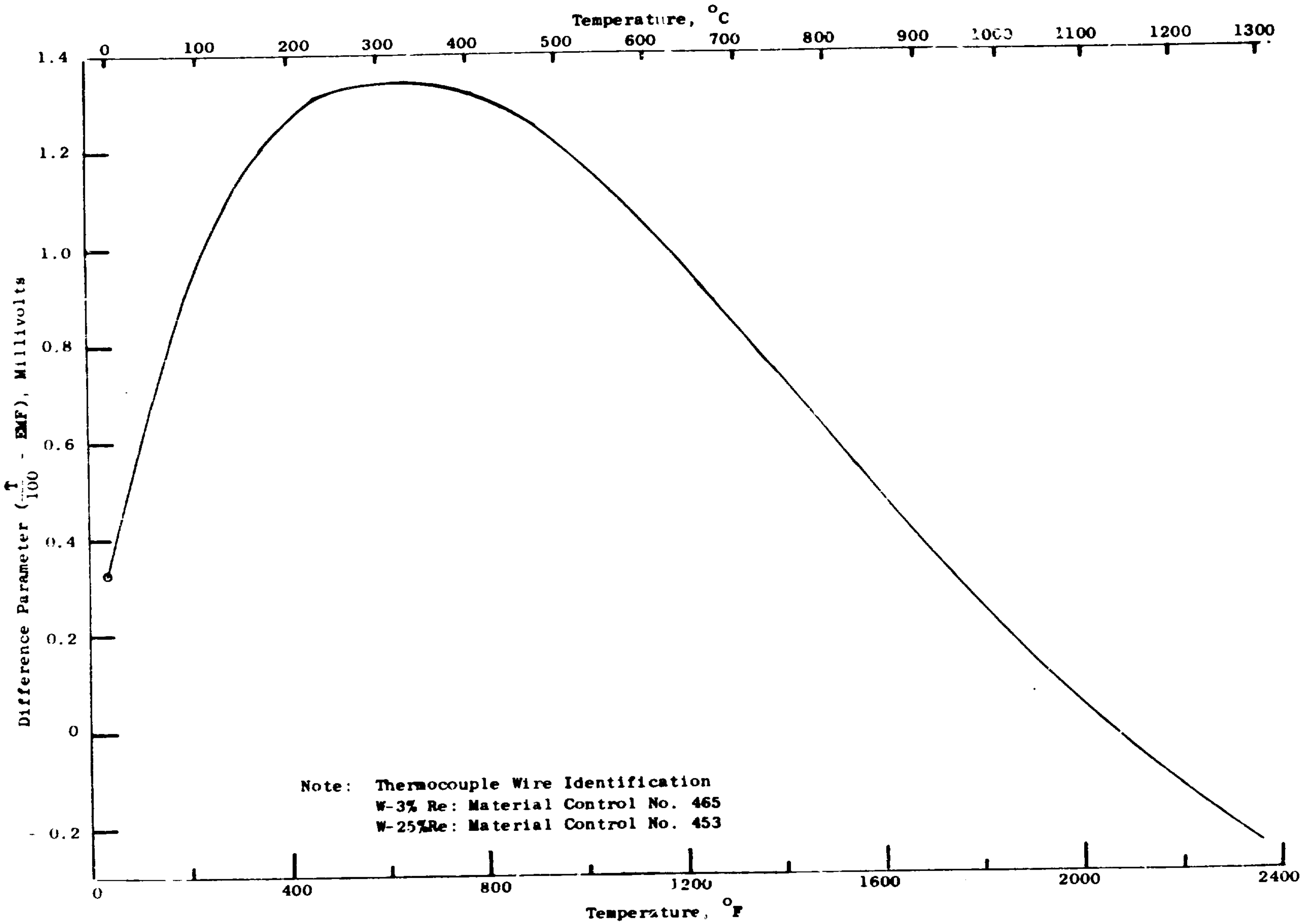

Figure I-3. Difference Parameter vs Temperature Obtained in Calibration of Two W-3Re/W-25Re Thermocouples Made From Wire Used to Instrument the T-111 Rankine System Corrosion Test Loop.

TABLE I-I

CALIBRATION DATA FOR W-3Re/W-25Re THERMOCOUPLE WIRE

Temperature °F	Temperature °C	Difference Parameter,[a] Millivolts	Total Output in Millivolts with 32°F (0°C) Reference Junction
32	0	0.320	0
100	37.8	0.587	0.413
160	71	0.790	0.810
200	93	0.908	1.092
250	121	1.028	1.472
300	149	1.126	1.874
350	177	1.196	2.304
400	204	1.253	2.747
450	232	1.298	3.202
480	249	1.315	3.485
510	266	1.324	3.776
610	321	1.338	4.762
670	354	1.339	5.361
730	388	1.330	5.970
790	421	1.313	6.587
830	443	1.296	7.004
900	482	1.251	7.749
950	510	1.215	8.285
1000	538	1.173	8.827
1100	593	1.073	9.927
1200	649	0.961	11.039
1300	704	0.849	12.160
1450	788	0.662	13.838
1650	899	0.416	16.084
1750	954	0.300	17.200
1850	1010	0.193	18.307
1950	1066	0.092	19.408
2050	1121	0.0	20.500
2150	1177	-0.086	21.586
2250	1232	-0.167	22.667
2350	1288	-0.240	23.740

[a] Difference Parameter = $\frac{T}{100}$ - EMF

TABLE I-II

COMPARISON OF W-3Re/W-25Re THERMOCOUPLE EMF WITH VALUES OBTAINED AT ENGELHARD INDUSTRIES, INC.

Temperature[a]		EMF Output, mv		
°F	°C	This Calibration	Engelhard[b]	Difference, mv
32	0	0.0	0.0	0.0
100	37.8	0.413	0.386	0.027
200	93	1.092	1.058	0.034
300	149	1.874	1.824	0.050
400	204	2.747	2.674	0.073
500	260	3.679	3.596	0.083
600	316	4.663	4.563	0.100
700	371	5.666	5.579	0.087
800	427	6.691	6.649	0.042
900	482	7.749	7.745	0.004
1000	538	8.827	8.843	-0.016
1100	593	9.927	9.957	-0.030
1200	649	11.039	11.084	-0.045
1300	704	12.160	12.220	-0.060
1400	760	13.279	13.362	-0.083
1500	816	14.399	14.504	-0.105
1600	871	15.522	15.638	-0.116
1700	927	16.642	16.769	-0.127
1800	982	17.753	17.895	-0.142
1900	1038	18.857	19.005	-0.148
2000	1093	19.954	20.106	-0.152
2100	1149	21.043	21.194	-0.151
2200	1204	22.126	22.273	-0.147
2300	1260	23.203	23.318	-0.115

(a) Based on International Practical Temperature Scale, 1948.

(b) Engelhard Industries, Inc., Research and Development Division - Final evaluation of November 1, 1965.

APPENDIX J

FAILURE MODE AND EFFECT ANALYSIS

The purpose of a failure mode analysis was to achieve a reliable system design through a systematic, qualitative approach that provides a means of taking preventative action in advance of the actual construction or operation of the system. The analysis encompasses the materials, components, and support equipment of the system and attempts to define the possible modes of failure or malfunction which would jeopardize the test and the action taken to improve the reliability of the system. Also given in this Appendix is a description of the principal loop components and of the test protection system.

1. PRINCIPAL LOOP COMPONENTS

In the primary circuit, the lithium is discharged from the EM pump and flows through an electrical resistance heater to a tube-in-tube counterflow boiler where heat is exchanged to the potassium of the secondary circuit. The lithium flow rate is measured by a permanent magnet flowmeter as it leaves the boiler on its return to the inlet of the EM pump.

In the boiling and condensing secondary loop, the potassium is discharged from the EM pump through a permanent magnet flowmeter and into the preheater where the temperature of the potassium is increased to its saturation temperature, approximately 2000°F (1093°C). It then flows into the tube-in-tube counterflow boiler. The liquid potassium is converted to vapor with 100°F (56°C) superheat and passes through the first nozzle and impinges upon the test blade specimen. The superheater vapor passes through the crossover section where it rejects heat by radiation to the chamber wall to lower its quality to 88 percent. The vapor then passes from stages 2 to 10 of the turbine simulator to the finned tube radiant condenser. The condenser fin is coated with iron titante to increase its emittance to 0.86. In the condenser the vapor is converted back to liquid and enters the subcooler reservoir before flowing into the EM pump.

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A description of the principal loop components follows:

Lithium Primary Loop

Lithium Pump

Type - Helical induction electromagnetic pump;

Mfg. - Medium A.C. Motor and Generator Dept., General Electric Company;

Model - 5KY414PB2;

Rating - 1.29 gpm (0.29 m^3/hr) at 100 psi (689 k N/m^2) head at 2200°F (1204°C);
5.0 gpm (1.13 m^3/hr) at 20 psi (138 k N/m^2) head at 2200°F (1204°C);

Duct Material - T-111;

Power Supply and Controls - 480-volt, 3-phase, 60-cycle power system - motor-operated autotransformer with manual or automatic control.

Lithium Flowmeter

Type - Permanent magnet;

Mfg. - Space Power and Propulsion Section, General Electric Company;

Magnet - 3000-gauss, Alnico V, stabilized and calibrated to 900°F (482°C);

Flow Tube - 0.375-inch (0.95-cm)-OD x 0.065-inch (0.16-cm) T-111 tube;

Insulation - 10 layers of dimpled Cb-1Zr foil, 0.002-inch (0.005-cm)-thick by 0.5-inch (1.2-cm)-wide;

Temperature - 2200°F (1204°C) maximum;

Accuracy - ± 8 percent (calculated.

Lithium Heater

Type - Electrical resistance, two 4-inch (10-cm)-diameter helical coils in series;

Tube Material - 0.375-inch (0.95-cm)-OD x 0.065-inch (0.16-cm)-wall T-111, total developed length of 101 inches (2.56 m);

Temperature - 2200°F (1204°C) maximum;

Power Supply - Combined temperature recorder and controller with a saturable reactor and stepdown current transformer, single-phase ac, 440-volt primary, 5-volt secondary;

Rating - 20 kva, 4000-ampere secondary.

Surge Tank

Size - 6 inches (15.2 cm) OD x 10 inches (25.4 cm) long;

Material - Cb-1Zr;

Tank Capacity - 234 in^3 (3840 cc);

Loop Capacity - 138 in.^{3} (2280 cc);

Inert Gas System

Gas - Ultrahigh-purity argon, 99.999 percent;

Regulator - Stainless steel, metal diaphragm;

Valves - Stainless steel, bellow seal;

Vapor Collector - Stainless steel cylinder 30-ml capacity;

Bimetallic Joint - Brazed Cb-1Zr to stainless steel;

Connecting Tube - 0.375-inch (0.95-cm) stainless steel TIG-welded construction.

Potassium Secondary Loop

Liquid Potassium Pump

Type - Helical induction electromagnetic pump;

Mfg. - Medium A.C. Motor and Generator Dept., General Electric Company;

Model - 5KY414 PFI;

Rating - 0.116 gpm (439 cm^3/min) at 200 psi (1380 kN/m^2) head at 1200°F (649°C);

0.290 gpm (1098 cm^3/min) at 150 psi (1032 kN/m^2) head at 1200°F (649°C);

Duct Material - T-111 tantalum alloy;

Power Supply - 480-volt, 3-phase, 60-cycle power system - motor-operated autotransformer with manual and automatic control.

Flow Control Valve and Isolation Valve

Type - 3/8-inch (0.95-cm) manual, bellows seal;

Model - Cb-442 MOD 3 (as modified by General Electric Company);

Mfg. - Hoke, Inc., Cresskill, New York;

Rating - 215 psi (1480 kN/m^2) at 1200°F (649°C);

Orifice Diameter - 5/32-inch (0.4-cm);

Material - All wetted parts are T-111 except the Cb-1Zr valve bellows and the Mo-TZM alloy plug. All seals are of welded construction. All threaded parts of valve assembly are refractory metal vs refractory metal or refractory metal vs stainless steel. Valve stem guide consists of a 0.125-inch (0.32-cm) pitch ball bearing screw assembly with 1/16-inch (0.16-cm) tungsten carbide balls.*

Potassium Flowmeter

Type - Permanet magnet;

Mfg. - Space Power and Propulsion Section, General Electric Company;

Magnet - 3000-gauss Alnico V, stabilized and calibrated to 900°F (482°C);

Flow Tube - 0.375-inch (0.95-cm)-OD x 0.065-inch (0.16-cm)-wall T-111;

Insulation - 10 layers of Cb-1Zr foil, 0.002-inch (0.005-cm)-thick by 0.5-inch (1.3-cm)-wide;

Accuracy - ± 8 percent calculated.

Pressure Transducer

Type - Stressed diaphragm;

Mfg. - Space Power and Propulsion Section, General Electric Company;

Material - T-111;

Recorder - Model 150, Sanborn Division, Hewlett Packard, Response - 10 millisecond full-scale;

Rating - Pressure - 0-235 psi (0-1620 kN/m^2),
Temperature - 0 to 800°F (-18° to 427°C),
Response - 10 milliseconds,
Accuracy - ± 1 percent full-scale;

Power Supply - Consolidated Controls Corporation, Bethel, Connecticut.

Pressure Transducer (5 required)

Type - Slack diaphragm;

Mfg. - Taylor Instrument Company, Rochester, New York;

Material - T-111 diaphragm and housing, stainless steel NaK-filled capillary and bourdon tube;

* Saginaw Steering Gear Division, General Motors, Saginaw, Michigan.

Rating - Pressure - 0-235 psi (0-1620 kN/m^2),

Temperature - 2000^{o}F (1093^{o}C),

Accuracy - $\pm$ 1 percent at 77^{o}F (25^{o}C),

Response - 1 second.

Recorder - Type HF, General Electric Company, response time - 1.5 seconds full-scale.

Potassium Preheater

Type - Electric resistance, two 4-inch (10.0-cm)-diameter helical coils in series;

Tube Material - 0.375-inch (0.95-cm)-OD 0.065-inch (0.16-cm) x 74-inch (188-cm)-long wall, T-111 alloy;

Electrode Material - Tantalum bar;

Temperature - 77^{o} to 2000^{o}F (25^{o} to 1093^{o}C);

Power Supply - Proportional temperature-controlled and silicon-controlled rectifier stepless power unit with a stepdown current transformer;

Rating - 5 kva, 1000 amperes.

Precision Wattmeter

Type 15 kilowatt wattmeter;

Ranges - 0 - 1500 watts,

0 - 4500 watts,

0 - 15,000 watts;

Current - 3000 amperes rms maximum;

Mfg. - Scientific Columbus, Inc., Columbus, Ohio.

Potassium Boiler and Superheater

Type - Tube-in-tube, counterflow with entrance plug;

Length - 250 inches (6.35 m) long arranged in a 10-inch (25.4-cm) helical coil;

Construction - Outer tube - 1.00-inch (2.5-cm) OD x 0.100-inch (0.25-cm) wall,

Inner tube - 0.375-inch (0.95-cm) OD x 0.065-inch (0.16-cm) wall,

Spacers - triform, spaced 60^{o} apart,

Plug - 18-inch (45.7 cm)-long, 1/16-inch (0.16-cm)-diameter wire helically wound (1-inch [2.54-cm] pitch) on a 1/8-inch (0.32-cm) rod;

Material - T-111;

Rating - 40 lb/hr (88 kg/hr) of potassium vapor at 2050^{o} ($1121^{o}C$) boiling temperature plus $100^{o}F$ ($56^{o}C$) superheat.

Turbine Simulator

Type - Fixed nozzle and blade assembly;

Material - Nozzle - molybdenum alloy TZC,

Blades - molybdenum alloy TZC and columbium alloy Cb-132M,

Housing - T-111;

Turbine Nozzle Stage Test Conditions*

Nozzle Number	Design Throat Velocity ft/sec	m/sec	Quality	Inlet Pressure, abs. psi	kN/m^2	Pressure Drop psi	kN/m^2
1	1000	305	$100^{o}F(56^{o}C)$S.H.	175	1222	-	-
2	|	|	86%	144	1005	21	145
3	|	|	to	117	816	27	186
4	|	|	88%	95	655	22	152
5	|	|	|	77	530	18	124
6	|	|	|	62	428	15	103
7	|	|	|	50	345	12	82
8	|	|	|	40	276	10	69
9	|	|	|	32	221	8	55
10	V	V	V	25	172	7	48

Inlet Temperature - $2150^{o}F$ ($1177^{o}C$);

Exit Temperature - $1400^{o}F$ ($760^{o}C$);

Flow Rate - 40 lb/hr (88 kg/hr);

Heat Rejection - Between stages 1 and 2 - 4000 Btu/hr (1.2 kw) radiantly rejected from 14.5 inches (36.8 cm) of uninsulated 1-inch (2.54-cm)-OD T-111 crossover tube.

Between stages 2 to 10 - 1300 But/hr (0.28 kw) radiantly rejected from turbine simulator shell with one layer of Cb-1Zr foil (ϵ = 0.2).

Potassium Vapor Condenser and Subcooler

Type - Finned, single-tube;

Material - T-111;

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* Property data used obtained from NRL Report 6128, August 1964.

Construction - Tube - 0.423-inch (1.07-cm)-ID drilled bar,

Fin - two 1/4-inch (0.64-cm)-thick x 4-inch (10.2-cm)-wide fins with Fe_2TiO_5 coated surface (ϵ = 0.86);

Length - 58 inches (147 cm);

Entrance Velocity - 350 ft/sec (106 m/sec);

Heat Rejection Control - Manually operated shutter assembly.

Subcooler Reservoir

Size - 2-inch (5.08-cm)-OD with 0.25-inch (0.64-cm)-wall x 4-inch (10.2-cm)-long;

Capacity - 6.2 in.^{3} (103 cc);

Material - T-111.

Surge Tank

Size - 6-inch (15.2-cm)-OD x 10-inch (25.4-cm)-long;

Material - Cb-1Zr:

Tank Capacity - 234 in.^{3} (3840 cc);

Loop Capacity - 139 in.^{3} (2282 cc).

Inert Gas System

Gas - Ultrahigh-purity argon, 99.99 percent;

Regulator - Stainless steel, metal diaphragm;

Valves - Stainless steel, bellows seal;

Vapor Collector - Stainless steel, 1.8-in.^{3} (30-ml) capacity;

Bimetallic Joint - Brazed Cb-1Zr to stainless steel.

Vacuum System

Vacuum Chamber

Size - Bell Jar - 128-inch (325-cm) high x 48-inch (122-cm) diameter,

Sump - 46-inch (117-cm) high x 48-inch (122-cm) diameter,

Spool - 24-inch (61-cm) high x 48-inch (122-cm) diameter;

Volume - 210 cu ft (5.94 m^3);

Ultimate Pressure - 5×10^{-11} torr (6×10^{-9} N/m^2);

Operating Pressure - 1×10^{-8} torr (1×10^{-6} N/m^2);

Bakeout Temperature - 500°F (260°C);

Gaskets - Copper seal.

Pumping System

Rough Pump - Type - Turbomolecular,

Model - 3102A,

Pumping Speed - 260 liter/second,

Blank-off Pressure - 2×10^{-9} torr (2×10^{-7} N/m^2),

Mfg. - Welch Scientific Company, Skokie, Illinois;

Ultrahigh-Vacuum Pump - Type - getter-ion,

Speed - 2400 liter/second,

Pressure - 1×10^{-2} (1 N/m^2) x 10^{-11} torr

([illegible] $\times 10^{-9}$ N/m^2);

Booster Pump - Type - Optically baffled titanium sublimation pump,

Number - 4,

Speed - 100,000 liters of hydrogen/second at

1×10^{-8} torr (1×10^{-6} N/m^2),

Operating Pressure - 10^{-2} to 10^{-11} torr (1 to 10^{-9} N/m^2);

Pressure Measurement

Total Pressure - Type - Nude ionization gauge,

Mfg. - Varian Associates, Palo Alto, California,

Range - 10^{-3} to 2×10^{-11} torr (10 to 2×10^{-9} N/m^2),

Accuracy - $\pm$ 6 percent;

Partial Pressure - Type - Permanent magnet partial pressure analyzer,

Mfg. - General Electric Company,

Pressure Range - 10^{-4} to 10^{-14} torr (10^{-2} to 10^{-12} N/m^2),

Mass Range - 2 to 50 amu,

Magnet - 3000-gauss, bakeable Alnico V.

2. TEST PROTECTION SYSTEM

The test loop control system includes safety circuits designed to protect the loop in the event of a malfunction of the control equipment, operator error or the loss of electrical or water service to the test area. The safety circuits listed below are checked during low-power test runs at the start of the test operations. After the final checkout, no safety circuit may be made inoperative or bypassed in subsequent test operations without permission of the Project Engineer or the Program Manager.

Loss of Cooling Water to Vacuum Chamber and Power Feedthrough

Loss of cooling water to the vacuum chamber facility can result in overheating and possible damage to the ceramic-metal seals of the electrical power feedthroughs and an increase in the chamber pressure due to outgassing of the chamber walls. In the event of a loss in the water flow, an electromechanical switch on the water supply inlet will interrupt the electrical power supply to the preheater and heater before permanent damage can occur.

Overtemperature of Primary Loop

If for any reason during loop operation, the maximum design temperature of the primary circuit is exceeded, an overtemperature circuit activated by the heater outlet temperature thermocouple will interrupt the electrical power to the preheater, heater, and EM pumps.

EM Pump Winding Overtemperature

The primary cause of an overtemperature of the stator of the EM Pump is the loss of cooling air which is required to remove the heat generated by I^2R losses in the stator winding. The air supply is delivered by a separate blower which can develop bearing or motor problems resulting in either total or partial loss in performance. In the event the windings do exceed their design temperature, a thermocouple attached to the windings activates a safety circuit which will interrupt the preheater, heater, and EM pump power.

Vacuum Chamber Leak

Endurance testing of refractory alloy, high-temperature alkali metal test loops requires a vacuum environment of 1×10^{-8} torr (1×10^{-6} N/m^2) or less to prevent the contamination of refractory alloy components. In the event of a large air leak or sudden increase in the chamber pressure, two independent safety circuits are activated to protect the loop from excessive oxidation.

The first circuit is activated when the chamber pressure exceeds 120 percent of the full-scale range of the ion gauge (usually set at 10^{-6} torr [10^{-4} N/m^2]). The ion gauge overpressure relay interrupts the electrical power to the EM pump, preheater, and heater.

The second safety circuit consists of two overpressure relays connected in series and operated by the ion pump current and the ionization gauge power supply. When the ion pump current increases to an equivalent pressure of 4×10^{-6} torr (5×10^{-4} N/m^2), the first relay of the safety circuit closes. If the pressure continues to rise to 4×10^{-4} torr (5×10^{-2} N/m^2), a second relay activated by the ionization gauge power supply is closed and complates a 110-v electrical circuit which fires an explosive valve and releases high-purity argon gas into the vacuum chamber to protect the loop from contaminating gases.

Partial Pressure Gas Analyzer

The residual gas in the vacuum chamber is intermittently scanned with a partial pressure gas analyzer during the test startup and subsequent test operation to determine its qualitative and quantitative composition. The gas analysis is usually taken daily during the steady-state operation but is made more frequently during the startup of the test. The partial pressure gas analyzer can be a most valuable tool in determining the source of the residual gas. By a comparative analysis of the residual gas composition with previous test runs, it can easily be determined if the gas source is the normal outgassing of the loop as the operating temperature is increased, a water leak in the vacuum chamber cooling system, an air leak in the vacuum chamber feedthroughs, or back-streaming from the ion pump.

The partial pressure gas analyzer can be used as an extremely sensitive leak detector to pinpoint leaks in either the feedthroughs or flange joints. Helium gas isusually used in leak checking the vacuum system although other gases can be used.

3. FAILURE MODE ANALYSIS

The general purpose of the failure mode analysis was to evaluate the design and proposed operation of the Corrosion Loop (T-111) System in advance of fabrication to elucidate the possible ways the components could malfunction or fail. This qualitative analysis provdes a means of making changes in the design or operation before the initiation of testing and thereby increasing the reliability of the system.

The primary objective of the test loop is the metallurgical evaluation of refractory alloys in a boiling and condensing potassium environment which will simulate projected space electric power systems. The structural design approach was therefore very conservative in the allowable stress of the alkali metal tubes and weldments. Special emphasis was placed in the preparation of a comprehensive material and process specification* for the procurement of refractory alloys and a specification for the procurement and handling of alkali metals. A quality control plan** was also issued for the control of raw materials and test components during the fabrication of the test loop.

The failure mode analysis was performed on components of the Corrosion Loop I (T-111) System under three (3) major categories namely:

A. Alkali Metal Purification and Handling Equipment;

B. Corrosion Loop I (T-111);

C. Auxiliary Test Equipment.

The Failure Mode Analysis sheets enclosed identify each component with its function and operational requirements.

Failure Mode - describes the possible ways the component could fail.

Method of Detection - the indication that shows a failure or a malfunction has occurred.

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* Potassium Corrosion Test Loop Development, Topical Report 3, Material Specification for Advanced Refractory Alloys, NASA CR-54761.

** Quality Control Manual Advanced Refractory Alloy Corrosion Loop Program, GE 08.100.01, Rev. No. 1.

__Failure Effect__ - the effect that failure has on the operation of the next higher assembly and possible effect upon the operation of the overall test system.

__Action Taken to Improve Reliability__ - the steps that have been taken in the design or proposed operation of the components to reduce the probability of a failure or a malfunction.

FAILURE MODE ANALYSIS SHEETS

a. ALKALI METAL PURIFICATION AND HANDLING EQUIPMENT

1. Lithium Hot Trap
2. Lithium High-Vacuum Distillation System
3. Lithium Transfer System
4. Alkali Metal Sampling System
5. Potassium High-Vacuum Distillation System
6. Potassium Hot Trap and Transfer System

FAILURE MODE ANALYSIS

COMPONENT LITHIUM HOT TRAP (a-1)		FUNCTION Purification of Lithium by Gettering of Nitrogen	
DWG NO. 263E821	NEXT HIGHER ASSY LITHIUM PURIFICATION SYSTEM	OPERATIONAL REQUIREMENTS Getter 35 lbs (16 kg) of Lithium at 1500°F (816°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Weld failure	Loss of argon pressure, smoke and fire.	Damage to equipment and possible injury to personnel.	Equipment designed to operate at essentially zero stress condition. Equipment leak checked before filling with lithium.
Valve bellows rupture	Same as above	Same as above	Limit valve temperature to 700°F (371°C) and 15 psi (103 kN/m^2).
Titanium liner failure	Inspection by disassembly	Slight corrosion attack of 316 SS body.	No action taken. Subsequent distillation will remove any metallic elements which might be picked up.

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FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
LITHIUM HIGH-VACUUM DISTILLATION SYSTEM (a-2)		Purification of Lithium by Distillation	
DWG NO. 246R608	**NEXT HIGHER ASSY** LITHIUM PURIFICATION SYSTEM	**OPERATIONAL REQUIREMENTS** High Vacuum Distillation at 1300°F (704°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Pot weld failure, valve bellows failure, bimetallic joint failure.	Lithium leak	Damage to equipment and possible injury to operating personnel.	Safety shields added to protect equipment and personnel.
Overheating of condenser and receiver.	Abnormal temperature rise above design operating temperature.	Damage to high vacuum system and bimetallic joints.	Maintain constant surveillance by qualified personnel during all distillation operations.
Ion-gauge rupture.	Air leak accompanied by lithium smoke.	Same as above.	Operate with ion gauge valved out of system except for pressure data measurements.
Cb-1Zr receiver rupture.	Lithium leak.	Damage to equipment and possible injury to personnel.	Safety shields added to protect equipment and personnel.

COMPONENT		FUNCTION	
LITHIUM TRANSFER SYSTEM (a-3)		Transfer Lithium from Still to Loop	
DWG NO. 263E823	**NEXT HIGHER ASSY** CORROSION LOOP I	**OPERATIONAL REQUIREMENTS** Zero External Leakage at 500°F (260°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Structural failure.	Lithium leakage.	Damage to equipment or injury to operating personnel.	Safety shields added to protect equipment and personnel. Fill operation made at lowest possible pressure and temperature.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
ALKALI METAL SAMPLING SYSTEM (a-4)		Obtain Samples of Alkali Metal from Test Loop	
DWG NO. 941D914	**NEXT HIGHER ASSY** CORROSION LOOP	**OPERATIONAL REQUIREMENTS** Zero External Leakage to 500 F (260°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Viewport failure.	Air leak with lithium smoke or fire.	Loss of sample and possible damage to sampling device.	Sample of lithium will be taken at the minimum temperature (less than 400 F [204°C]).

COMPONENT		FUNCTION	
POTASSIUM HIGH VACUUM DISTILLATION SYSTEM (a-5)		Purification of Potassium by Distillation	
DWG NO. 246R820	**NEXT HIGHER ASSY** POTASSIUM PURIFICATION SYSTEM	**OPERATIONAL REQUIREMENTS** Zero External Leakage at 700°F (371°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Component leak	Loss of vecuum plus potassium smoke or fire.	Loss of potassium charge and possible damage to equipment and operating personnel.	Safety shields and protective clothes used during hazardous operation.

COMPONENT		FUNCTION	
POTASSIUM HOT TRAP AND TRANSFER SYSTEM (a-6)		Purification and Transfer of Potassium Charge to Loop	
DWG NO. 246R611	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS** Zero Bxternal Leakage at 500°F (260°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Structural failure.	Potassium leakage.	Damage to equipment or injury to operating personnel.	Safety shields added to protect equipment and personnel. Fill operation made at lowest possible pressure and temperature.

FAILURE MODE ANALYSIS SHEETS

b. CORROSION LOOP I

1. Lithium EM Pump Duct Assembly
2. Potassium EM Pump Duct Assembly
3. I^2R Lithium Heater
4. I^2R Potassium Preheater
5. Pressure Transducer (Slack Diaphragm)
6. Pressure Transducer (Stressed Diaphragm)
7. Potassium Metering Valve
8. Potassium Isolation Valve
9. Boiler Assembly
10. Turbine Simulator
11. Potassium and Lithium Surge Tanks
12. Condenser-Subcooler
13. Argon Pressurization System
14. Permanent Magnet Flowmeter
15. Condenser Shield

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
LITHIUM EM PUMP DUCT ASSEMBLY (b-1)		Circulate Liquid Lithium in Primary Heater Loop	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
941D881	CORROSION LOOP	5 gpm (1.13 m^3/hr) of Lithium at 2100°F (1149°C) for 10,000 Hours	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Weld failure.	Loss of lithium with deposition on cold surfaces including vacuum chamber viewport.	Degradation of heat circuit performance.	Compatibility study of lithium and T-111 for welded specimens.
	Erratic signals from loop thermocouples, ion gauge and partial pressure gas analyzer	Operation and control of the loop could be affected by false signals generated from shorted lead wires.	100% in-process helium leak check and radiographic inspection for critical applications.
Structural failure.	Same as above.	Same as above.	Completed 5000-hr component evaluation test at 2150°F (1177°C) at 200 psia (1378 kN/m^2).
Flow blockage in helical passages by mass transfer.	Decrease in pump performance.	Reduction in heat transfer capacity of lithium circuit.	Lithium purification procedures adopted to minimize mass transfer due to lithium impurities acting as carriers. Loop operation procedures require flushing of loop and sampling of lithium to insure impurity level is below safe operating limit.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION
POTASSIUM EM PUMP DUCT [illegible] Y (b-2)		Boiler Feed Pump in Potassium Loop
DWG NO. 263E643	NEXT ASSY CORRO[illegible] LOOP	OPERATIONAL REQUIREMENTS 40 lb/hr (18 kg/hr) of $1200^{o}F$ ($6[illegible]^{o}C$) Potassium with 200 psi (1047 kN/m^2) Pressure Head

(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Weld failure.	Loss of potassium with deposition on cold surfaces including vacuum chamber viewport.	Degradation of boiler performance for severe leak.	Compatibility study of potassium and T-111 for welded specimens.
		Test shutdown for weld repair	100% in-process helium leak check and radiographic inspection for critical applications.
	Erratic signals from loop thermocouples, ion gauge and partial pressure gas analyzer	Operation and control of the loop could be affected by false signals generated from shorted lead wires.	
Structural failure.	Same as above.	Same as above.	Completed 5000-hr prototype loop test at $1200^{o}F$ ($649^{o}C$) at 150 psi (1033 kN/m^2).
Flow blockage in helical passages by mass transfer or foreign particles.	Decrease in pump performance.	Degradation of the boiling and condensing potassium loop.	Added particle trap between condenser and pump. Loop operation procedures require flushing of loop and sampling of potassium to insure impurity level is below safe operating limit.
Vapor lock.	Loss of potassium flow.	Overtemperature of preheater and heater loop.	Redesigned the prototype pump to minimize cavitation at the inlet which was observed in the prototype test.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
I^2R LITHIUM HEATER (B-3)		Electric Heater for Lithium Loop	
DWG NO.	NEXT HIGHER ASSY	OPERATIONAL REQUIREMENTS	
263E829	CORROSION LOOP	10 kw at 2250°F (1232°C) for 10,000 Hours	

(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Electrical short between helical coils.	Fluctuations in heater voltage and current.	Damage to tube wall by electrical discharge.	Spacing between coils increased.
Fatigue failure of heater coils due to induced mechanical vibrations from AC current.	Visual observation of coils through vacuum chamber viewport.	Loss of lithium and degradation of heater circuit performance.	No serious vibrations observed in 5000-hr prototype test at similar test conditions.
Overheating due to loss of lithium flow.	Temperature excursion.	Possible grain growth in tube wall due to high temperature excursion.	Overtemperature safety circuit added to interrupt electric power input. Current limiter installed in heater power supply.

FAILURE MODE ANALYSIS

COMPONENT I^2R POTASSIUM PREHEATER (b-4)	**FUNCTION** Preheater Potassium Boiler Feed to Saturation Temperature
DWG NO. 941D884 — **NEXT HIGHER ASSY** CORROSION LOOP	**OPERATIONAL REQUIREMENTS** Preheat 40 lb/hr (18 kg/hr) of Potassium Liquid to 2050°F (1121°C)

(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Electrical short between helical coils.	Fluctuation in preheater voltage and current.	Damage to tube wall by electrical discharge.	Spacing between coils increased.
Fatigue failure of heater coils due to induced mechanical vibrations from AC current.	Visual observation of coils through vacuum chamber view-port.	Loss of potassium and degradation of boiler circuit performance.	No serious vibrations observed in 5000-hour prototype test.
Overheating of tube due to loss of potassium flow.	Temperature excursion.	Material property change in preheater tube due to high temperature excursion and unstable loop operation.	Current limiter installed in preheater power supply.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
PRESSURE TRANSDUCER (SLACK DIAPHRAGM) (b-5)		Measure Vapor or Liquid Potassium Pressure	
DWG NO.	NEXT HIGHER ASSY	OPERATIONAL REQUIREMENTS	
263E826	CORROSION LOOP	Measure 0-30 cps Pressure Fluctuations in the 0-200 psia (1378 kN/m^2) Range	

(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Rupture of slack diaphragm	Decrease in response time.	Diffusion of NaK into potassium loop. Reduction in sensitivity of pressure transducer.	Added process tube extension to all pressure transducers to reduce operating temperature to less than 800°F (427°C).
Weld or bimetallic joint failure	Decrease in response time. Deposition of NaK on vacuum chamber viewport. Erratic signals from loop thermocouples and ion gauge.	Change calibration of pressure transducer. Test shutdown for weld repair.	100% in-process helium leak check and radiographic inspection for critical applications.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
PRESSURE TRANSDUCER (STRESSED DIAPHRAGM) (b-6)		Measure High Speed Pressure Fluctuations	
DWG NO. 119C2992	NEXT HIGHER ASSY CORROSION LOOP	OPERATIONAL REQUIREMENTS Measure 100 cps Pressure Fluctuations at 800 F (427^{o}C)	
(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Exceed yield point of stress diaphragm.	Loss of sensitivity or zero shift.	Inability to measure absolute pressure of loop.	Redesign diaphragm to increase load capacity.
Rupture of stress diaphragm.	Deposition of potassium on vacuum chamber viewport.	High frequency pressure fluctuations could not be detected.	Backup plate installed behind stress diaphragm to limit the amount of creep.
	Loss of sensitivity.		

COMPONENT		FUNCTION	
POTASSIUM METERING VALVE (b-7)		Regulate the Potassium Flow Rate for Stable Operation	
DWG NO. 263E827	NEXT HIGHER ASSY CORROSION LOOP	OPERATIONAL REQUIREMENTS Regulate 40 lb/hr (18 kg/hr) of Potassium at 1200^{o}F (649^{o}C)	
(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Rupture of valve bellows.	Deposition of potassium on vacuum chamber viewport.	Loss of potassium inventory.	Limit operating temperature to 1200 F (649^{o}C).
Galling or self-welding of valve actuation system.	Increase in torque required to operate valve.	Loss of flow control.	Bearing surfaces changes to refractory alloys.
Blockage of flow area between plug and seat by particulate matter in the flow circuit.	Increase in pressure drop across valve.	Adjustment required to maintain design flow rate.	Trap installed at exit of condenser to collect particulate matter.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
POTASSIUM ISOLATION VALVE (b-8)		Isolate Potassium Surge Tank from Loop	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
263E827	CORROSION LOOP	On-off Operation with Liquid Potassium at 500 F (260°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Rupture of valve bellows.	Deposition of potassium on vacuum chamber viewport.	Loss of potassium inventory.	Limit operating temperature to 1200 F (649°C).
Galling or self-welding of valve actuation system.	Increase in torque to operate valve.	Inability to dump loop if valve is in closed position.	All bearing surfaces changed to refractory alloys.
Internal leakage.	Change in loop operating conditions.	Increase sensitivity to loop instability.	Six inches (15.2 cm) added to boiler plug to improve loop stability.

COMPONENT		FUNCTION	
BOILER ASSEMBLY (b-9)		Tube-in-Tube, Counterflow Lithium-Potassium Boiler	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
263E828	CORROSION LOOP	Vaporize and Superheat 40 lb/hr (18 kg/hr) of Potassium to 2150°F (1177°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Leak in center tube	Chemical analysis of potassium at the conclusion of test.	Lithium leak in potassium loop.	100% in-process leak check and radiographic examination
	Change in loop operating characteristics.	Change saturation pressure and temperature in boiling and condensing loop.	
Leak in outer tube.	Deposition of lithium on vacuum chamber viewport.	Loss of lithium.	100% in-process leak check and radiographic examination.
Detachment of plug segments from boiler entrance.	Change in boiler pressure drop and temperature profile.	Unstable loop operation or even complete blockage of turbine simulator.	Plug design checked in 5,000-hr prototype test.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION
TURBINE SIMULATOR (b-10)		Test Nozzle and Blade Specimens at Potassium Turbine Test Conditions
DWG NO. 119C2929 941D874	**NEXT HIGHER ASSY** CORROSION LOOP	**OPERATIONAL REQUIREMENTS** Operate at 2150°F (1177°C) for 10,000 Hours with a Maximum Potassium Vapor Velocity of 1000 ft/sec (305 m/sec).

(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Erosion of blade or nozzle test specimens.	Change in pressure drop across the turbine simulator.	Change in loop operating characteristics.	No action taken - purpose of test is to evaluate the effect or high velocity, wet potassium vapor on test nozzles and blades.
Mass transfer deposits on the nozzle throat.	Same as above.	Same as above.	Same as above.
Weld failure.	Loss of potassium and deposition on vacuum chamber viewport erratic signals from loop thermocouples and ion gauge.	Test shutdown for weld repair.	100% in-process leak check and radiographic inspection of all welds.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
POTASSIUM AND LITHIUM SURGE TANKS (b-11)		Liquid Potassium and Lithium Storage Tank	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
941D869	CORROSION LOOP	Store Excess Liquid Metal Inventory at 350 F (177°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Weld failure by argon over-pressurization	Loss of potassium or lithium. Deposition on vacuum chamber viewport erratic signals from loop thermocouples and ion gauge.	Test shutdown for weld repair.	Argon supply regulator set at 250 psi (1722 kN/m²) maximum. Argon supply line isolated from loop by shut-off valve.
Flow blockage in dip leg.	Difficulty in filling or dumping loop.	Serious if blockage is complete.	Tube end angled at 45° to increase flow area. Liquid metal charge is passed through 5 micron filter to remove particulate matter during fill operation.

COMPONENT		FUNCTION	
CONDENSER-SUBCOOLER (b-12)		Condense Potassium Vapor and Sub-cool Potassium Liquid	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
263E825	CORROSION LOOP	Condense 40 lb/hr (18 kg/hr) of Potassium Vapor at 1400°F (760°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Weld failure.	Loss of potassium deposition of potassium on vacuum chamber viewport.	Test shutdown for weld repair.	100% in-process helium leak check and radiographic inspection of all welds.
Spalling or deterioration of iron titanate coating.	Change in temperature distribution in condenser.	Change in liquid-vapor interface position in condenser tube.	No action taken. Previous tests indicate coating is reliable at design temperature.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
ARGON PRESSURIZATION SYSTEM (b-13)		Evaluate and Pressurize Loop with High Purity Argon	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
263E825	CORROSION LOOP	Operate from 0-200 psia (1378 kN/m^2) at 500°F (260°C) Maximum	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Plugging of gas line by solidified alkali metal vapor condensate.	Loop pressure does not respond to changes in supply pressure.	Loop operating pressure cannot be changed.	Helical vapor condenser and liquid collector added to the argon pressurization line.
Plugging of gas line by over-filling surge tank during fill operation.	Same as above.	Same as above.	Fill procedures changed to eliminate blowdown of charge pot which resulted in plugging of prototype loop gas pressurization line.

COMPONENT		FUNCTION	
PERMANENT MAGNET FLOWMETER (b-14)		Measure Potassium and Lithium Flow Rate	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
246R897	CORROSION LOOP	Operate at Liquid Metal Temperatures of 2200°F (1204°C)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Electrode weld failure due to stress caused by thermal expansion.	Loss of flow signal.	Flow rate must be calculated by calorimetric methods.	Expansion loops added to electrode leads to relieve stress due to thermal expansion.
Flow tube in contact with the support structure.	Erratic flow signals.	Same as above.	Flow meter magnet gap aligned with the predicted movement of flowmeter tube during start-up.
Magnet flux change.	Decrease in flow sensitivity.	Same as above.	Thermal insulation added to reduce heat flux from high temperature components to limit magnet temperature to 850°F (454°C).

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
CONDENSER SHIELD (b-13)		Regulate Heat Rejection Rate from Potassium Condenser	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
119C276[illegible]	CORROSION LOOP	Maintain Vapor Liquid Interface in Condenser	

(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Mechanical failure in gears bearings or cable drive.	Visual observation through vacuum chamber viewport.	Result in off-design operation of condenser.	Design checked in 5000-hr prototype test - no further action required.

FAILURE MODE ANALYSIS SHEETS

c. AUXILIARY TEST EQUIPMENT

1. Vacuum Chamber System
2. Electrical Power Feedthrough
3. Loop Support System
4. Heater Power Supply
5. Preheater Power Supply
6. EM Pump Power Supply
7. Argon Flooding System
8. Thermocouples

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
VACUUM CHAMBER SYSTEM (c-1)		Maintain Ultrahigh Vacuum Environment During Loop Operation	
DWG NO.	NEXT HIGHER ASSY	OPERATIONAL REQUIREMENTS	
Commercial	None	1×10^{-8} Torr (1.33×10^{-6} N/m^2) Vacuum for 10,000 Hours	

(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Air leak in flanges, ports and feedthroughs.	Pressure rise on ion gauge or partial pressure gas analyzer.	Loop safety circuit will shutdown test when pressure exceeds 4×10^{-6} torr (5×10^{-4} N/m^2). When the pressure exceeds 1×10^{-4} torr (1×10^{-2} N/m^2) the argon flooding system will be activated flooding the chamber with argon (c-7).	All metallic gaskets used in assembly of vacuum chamber.
Electrical shorting of ion pump cell.	Erratic ion pump current and increase in vacuum Chamber pressure.	Reduce pumping speed of ion pump.	Ion pumps cleaned and reconditioned before start of test.
Water leak in cooling channel.	Increase in percent water vapor content of residual gases.	Loop safety circuit will shutdown test when pressure exceeds 4×10^{-6} torr (5×10^{-4} N/m^2).	Filters installed in water supply system to trap particulate matter which can contribute to corrosion cracking of chamber wall. Cooling channels modified to eliminate air pockets which may contribute to possible corrosion.
Crack or breakage of glass viewports.	Pressure rise detected by ion gauge.	Possible contamination of loop components. Loop safety circuits will shut down test when pressure exceeds 4×10^{-6} torr (5×10^{-4} N/m^2).	Safety guards will be mounted over all viewports, when not being used. Access to the area in the vicinity of the sightports will be limited to personnel cognizant of the viewports.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
ELECTRICAL POWER FEEDTHROUGH (c-2)		Power Feedthroughs for Preheater and Heater	
DWG NO.	NEXT HIGHER ASSY	OPERATIONAL REQUIREMENTS	
Commercial Part	VACUUM CHAMBER	1000 Ampere Steady State Operation with Zero Leakage	
(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Ceramic to metal braze joint failure (mechanical).	Pressure rise on ion gauge.	Loop safety circuit will shut down test when pressure exceeds 4×10^{-6} torr (5×10^{-4} N m^{-2}). When the pressure exceeds 1×10^{-4} torr (1×10^{-2} N m^{-2}), the argon flooding system will be activated flooding the chamber with argon (c-7).	Flexible cable connections used to limit force on brazed joint.
Ceramic to metal braze joint failure (thermal).	Same as above.	Same as above.	Safety circuit added to shutdown loop in the event of loss of cooling water to power feedthroughs.
Ceramic to metal braze joint failure (Corrosion attack).	Same as above.	Same as above.	Filters installed in water supply system to trap particulate matter which could contribute to corrosion cracking of chamber wall. Cooling channels modified to eliminate air pockets which may contribute to possible corrosion.

FAILURE MODE ANALYSIS

COMPONENT LOOP SUPPORT SYSTEM (c-5)	**FUNCTION** Support Loop by Lever and Counter Weight System
DWG NO. / **NEXT HIGHER ASSY** CORROSION LOOP	**OPERATIONAL REQUIREMENTS** Accommodate Thermal Expansion Between Loop and Support Structure

(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Dislodgement of counter weights due to vibration induced rotation of retainer nuts.	Visual observation.	Damage to loop by impact of falling counterweights. Sagging or misalignment of loop components which could affect loop performance.	Retainer nuts secured by welding or pinning.
Buckling of support structure.	Same as above.	Same as above.	Reinforce support structure with cross braces for added stiffness.

COMPONENT HEATER POWER SUPPLY (c-4)	**FUNCTION** Regulate and Control Electrical Power to Sodium Heater
DWG NO. Commercial / **NEXT HIGHER ASSY** CORROSION LOOP	**OPERATIONAL REQUIREMENTS** 10 KW of Single Phase AC Power for 10,000 Hours

(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Electronic malfunction.	No control on power input.	Shutdown of test.	Saturable core reactor selected because of its reliability.
Main power supply failure.	All electric power off. Alarm at fire station.	Shutdown of test, no damage to system. Test can be restarted when power restored.	No action taken - commercial power supply has good record of dependability.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
PREHEATER POWER SUPPLY (c-5)		Regulate and Control Electric Power to Potassium Preheater	
DWG NO.	NEXT HIGHER ASSY	OPERATIONAL REQUIREMENTS	
[illegible]	CORROSION LOOP	5 KW Single-Phase AC Power for 10,000 Hours	
(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Electronic controller malfunction.	Change in operating conditions.	Unstable loop operation or possible test shutdown due to actuation of loop safety circuits.	Additional 6 in. (15 cm) added to plug length to improve the loop stability and attenuate pressure fluctuations during unstable operation.
Main power supply failure.	Electric power off. Alarm at fire station.	Interruption of test without deleterious effects on loop.	No action taken - Commercial power supply has good record of dependability. Vacuum chamber cooling water valve closed and bakeout heaters turned on to prevent the loop from freezing.

COMPONENT		FUNCTION	
EM PUMP POWER SUPPLY (c-6)		Control and Regulate Electric Power to EM Pump	
DWG NO.	NEXT HIGHER ASSY	OPERATIONAL REQUIREMENTS	
		Supply 5 to 15 KW of 3-Phase AC Power to EM Pump Stator	
(1) FAILURE MODE	(2) METHOD OF DETECTION	(3) FAILURE EFFECTS	(4) ACTION TAKEN TO IMPROVE RELIABILITY
Capacitor failure.	Reduction in pump performance.	An inability to maintain test conditions, requiring a test shutdown to replace capacitors and possibly power state.	No action required. Capacitor rating exceeds proposed requirement.
Overtemperature of stator windings.	Winding overtemperature alarm.	Shutdown test.	Oversize air blowers installed to cool stator windings.

FAILURE MODE ANALYSIS

COMPONENT		FUNCTION	
ARGON FLOODING SYSTEM (c-7)		Protect T-111 Loop Components from Severe Oxidation	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
Commercial	VACUUM CHAMBER SYSTEM	Flood Vacuum Chamber with Argon Gas if Pressure Exceeds 1×10^{-4} Torr (1×10^{-2} N/m^2)	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Malfunction in electronic control circuit.	Explosive valve fired. chamber filled with argon gas.	No deleterious effects. Loop shutdown to replace explosive valve.	Modified high pressure detection circuit to include confirmation of high pressure by ion gauge and ion pump current.
Argon instability.	Temporary argon pressure rise as detected by partial pressure gas analyzer.	Possible actuation of overpressure safety circuit.	Overpressure triggering relay set at 1×10^{-4} torr (1×10^{-2} N/m^2) which is a decade below maximum pressure rise observed during prototype corrosion loop operation.

COMPONENT		FUNCTION	
THERMOCOUPLES (c-8)		Measure Test Loop Operating Temperature	
DWG NO.	**NEXT HIGHER ASSY**	**OPERATIONAL REQUIREMENTS**	
		10,000-Hour Operation at 2300°F (1260°C) in 1×10^{-8} Torr (1×10^{-6} N/m^2) Vacuum	
(1) FAILURE MODE	**(2) METHOD OF DETECTION**	**(3) FAILURE EFFECTS**	**(4) ACTION TAKEN TO IMPROVE RELIABILITY**
Electrical short of lead wires.	Erratic or unreasonable signals based on predicated operating temperature.	Seriousness depends on number and location of thermocouple failures.	Redundant thermocouples added to required control areas.
Opening of thermocouple joint or lead wires.	Loss of signal and high electrical resistance	Same as above.	Provide thermal expansion loop near thermocouple junctions.
Calibration shift.	Change in measured loop operating temperatures.	Precise operating temperatures of loop components will be difficult to establish.	All thermocouple wires are thermally stabilized before installation.

APPENDIX K

REMOVAL, REPAIR, AND REPLACEMENT OF THE T-111 BOILER

As indicated in Section VIII, Pretest Operation, of this report, a leak between the potassium and lithium circuits in the boiler was discovered during the initial attempt to reach the test conditions. Considerable effort was expended in the analysis of the problems which required shutdown of the loop and the subsequent actions taken to put the loop back into operation without jeopardizing the objectives of the test program. The repair of an all-refractory alloy loop after exposure to alkali metal had not previously been demonstrated anywhere successfully. Many new techniques had to be developed to accomplish this task. The work performed is described in considerable detail since similar techniques could be employed in repair or replacement of sections of any refractory alloy - alkali metal system. Such techniques were subsequently utilized in the replacement of a test section in the 1900°F Lithium Loop which was also part of this contract (NAS 3-6474).

1. Techniques Employed to Drain and Analyze the Alkali Metals from the Loop

Following the plugging problems which caused loop shutdown, as described in Section VIII, a sampling and distillation system was designed and fabricated to gravity-drain the potassium from the surge tank in a manner to trap any particulate matter for subsequent analysis. This system, shown schematically in Figure K-1, was attached to the loop system at the potassium fill valve (KE). The sample tube consisted of a 36-inch (91-cm) length of 1/2-inch-OD x 0.020-inch-thick-wall (1.27-cm-OD x 0.050-cm-thick-wall) Type 321 stainless steel tubing and was connected, as shown in Figure K-1, to assure that the sample tube would be filled during the draining operation. The transfer line, connecting the sampler to valve KK and the still pot fill valve, was made of 1/2-inch-OD x 0.060-inch-thick-wall (1.27-cm-OD x 0.152-cm-thick-wall) Type 321 stainless steel tubing.

The still was designed with a "filings' shield," such that the top could be cut off without introducing metal filings into the still. The still pot had a capacity of 4800 cc and the receiver had a capacity of

Notes: 1. All Joints Welded and Helium Leak Checked

2. All Valves Hoke TY445 or HY473A - Welded Bellows and Positive Return

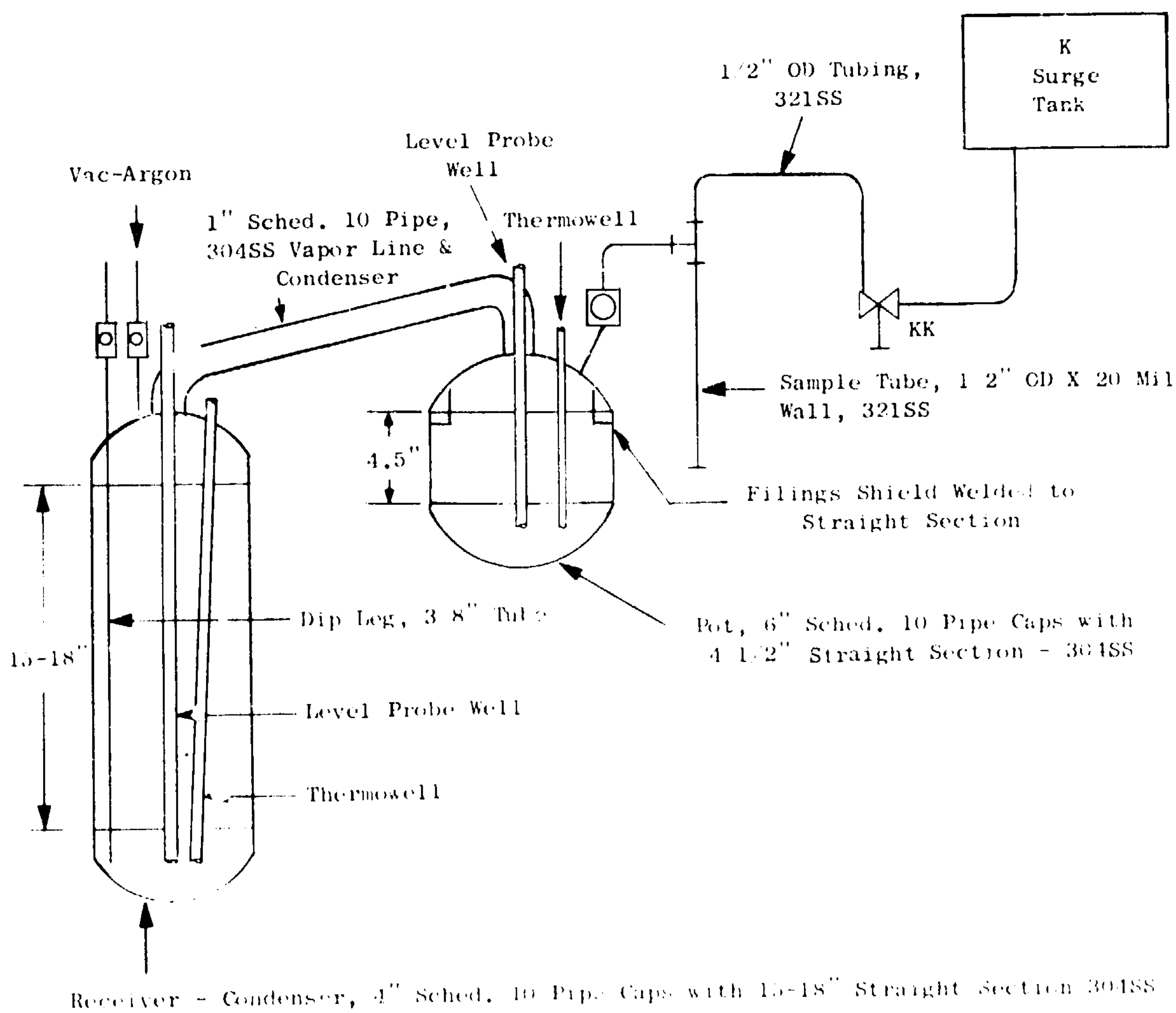

Figure K [illegible]. Distillation and Sampling System Used in Isolating Particulate Matter in the Potassium Drained From the T 111 Corrosion Test Loop.

5100 cc. All valves were welded, bellows-sealed, Type 316 stainless steel. Prior to draining the potassium from the surge tank into the still, the still system was baked out at temperatures from 200° to 600°F (93°C to 316°C) until the pressure rise rate was less than one micron-liter per minute.

The potassium was subsequently drained and sampled. The potassium surge tank was first pressurized with argon to force the potassium into the loop. The EM pump was turned on and an immediate, but low, potassium flow rate was established in the forward flow direction (the loop was previously shut down with the potassium loop completely plugged). The loop was immediately dumped into the surge tank and then refilled with a marked improvement in the potassium circulation. The process of filling, circulating, and dumping the loop was repeated five times with a 30-minute dwell time in the surge tank to allow any particulate matter which might be present to settle out in the surge tank. After the fifth dump, the potassium was drained from the surge tank as described below.

The line from valve KK (See Figure K-1) to the loop surge tank was at 350°F to 400°F (177°C to 204°C). The drain system was evacuated to less than one micron back to valve KK. Valve KK was cracked and flow began as indicated by a sharp temperature rise in the line between valve KK and the surge tank (from 350° to 450°F, 177° to 232°C) and by the level probe in the still pot. Valve KK was then completely opened and the alkali metal drained from the surge tank until the MSAR inductive-type level probe in the still pot indicated 600 to 700 cc of potassium, at which time flow ceased. Valve KK was then closed, the drain system was pressurized, and the sampler was held under 25 psia (1.72×10^5 N/m^2) pressure at a temperature above the potassium melting point overnight and then cooled to room temperature. All power was shut off on the drain system, and all lines from the still pot to valve KK were radiographed to determine if a plug in the lines could be located. The radiographs showed nothing but a few voids in the alkali metal in the sample tube.

The potassium was then distilled from the pot into the receiver for a period of about 66 hours at temperatures of 600°-700°F (316°-371°C). The level probe indicated no change in the level of metal in the receiver for a period of at least 16 hours before distillation was stopped.

Following distillation, the potassium drain system and still pot were removed by crimping the line above valve KK, cutting the line between valve KK and the crimp, and capping both ends of the cut line with Swageloks. The entire still-drain system was then placed in the welding chamber, and the sampler and drain line was cut between the still pot and the valve shown in Figure K-1, under high-purity helium. The drain line - sampler system was then removed from the chamber for analysis of the contents of the lines, the results of which will be discussed later.

The still pot was then cut open to investigate any residue remaining in the bottom of the pot. The entire cutting operation was performed in the high-purity helium environment of the welding chamber. The still pot was cut open with a hacksaw just above the bottom of the filings' shield shown in Figure K-1.

Inspection of the bottom of the still pot revealed black, particulate matter and a thin film of residual lithium. Most of the black particles were swept into a glass vial using a brush, and the remainder were sucked into an Erlenmeyer flask. The lithium was scraped out and placed in another glass vial, and the vials were sealed under helium.

A number of specimens were designated as appropriate for analytical chemical, spectrographic, or X-ray examination. They are identified and the results are given in Table K-I. The significance of the results will be discussed below.

Prior to the final shutdown, it was conjectured that the plugging of the metering valve was due to particulate matter and that the boiling instabilities could possibly have been intensified by lithium in the potassium resulting from a leak between the primary and secondary circuits. A potassium sample (No. 1622) was removed from the potassium transfer line between valve FF and KK (similar to that shown in Figure K-2). The emission spectrographic results shown in Table K-1 indicate that all metallic impurities except lithium were at or below the detection limits. A flame photometric analysis for lithium indicated 40 ppm. This should be compared with the flame photometric result of < 10 ppm lithium for sample No. 1487A removed from the charge pot prior to filling the loop; and also with the emission spectrographic result of < 2 ppm lithium for

TABLE K-I

CHEMICAL ANALYSIS OF THE ALKALI MET

T-111 RANKINE SYSTEM CORROSION TES

Sample Number and Date	Identity	Analytical[a] Lab	ppm in the Metal O	C	N	% Li	% K	Ag	Al	B	Ba	Be	[illegible]	[illegible]	[illegible]	Cr	[illegible]	Fe
292 1-4-66	Lithium As received	GE		13[illegible] 158	801 834 871			<5	<5			<5	15[illegible]	<25	<5	<5	[illegible]	<5
		GGA (FN)[b]	130 ± 25 130 ± 25															
1588 3-25-68	Lithium As received Resampled	GE (D)	278															
293 1-4-66	Lithium After filtering at 400°F through a 5-micron filter into the hot trap	GE		97 101	755 77[illegible] 841			<5	<5			<5	5[illegible]	<25	<5	[illegible]	[illegible]	<5
		GGA (FN)	150 ± 28 150 ± 28															
309 4-4-66	Lithium After hot trapping at 1500°F for 126 hours. Sampled at 500°F	GE			11 10 5 3			<5	5	<50		<5	5	<25	<5	<5	<5	5
		GGA (FN)	106 ± 16 123 ± [illegible]															
[illegible]020 4-20-67	Lithium Residue dissolved out of 15-ml hot filter which plugged while trying to return lithium from receiver to hot trap after overflowing still pot into receiver. Metals reported as ppm in the residue	GE						[illegible]	50	5		50	[illegible]	Nil	Nil	[illegible]	[illegible]	200
[illegible] 5-4-67	Lithium After distilling 15 lbs in [illegible] lb batches. The first two batches were returned to the hot trap and the third batch was sampled at 385°F from the receiver. No filter present at this time	GE		[illegible] 42	[illegible]			[illegible]	[illegible]	<50	<50	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		GGA (FN)	245 ± [illegible]															
[illegible]061 [illegible]	Lithium Second sample from [illegible] lb batch (See Sample [illegible]) [illegible]	GE		[illegible] 16 55	[illegible] 22													
		GGA (FN)	[illegible] ± 4															
[illegible] [illegible]	Lithium Third sample from [illegible] lb batch (See Samples [illegible] & [illegible]064) Sampled at [illegible]	GE			20 7													
[illegible] [illegible]	Lithium Sampled at 42[illegible]°F from still receiver after distilling for the T-111 [illegible]	GE		26	[illegible]			[illegible]	[illegible]	<50	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
		GGA (FN)[b]	273 ± [illegible]															
		ORNL (FN)	42 [illegible]															
		ORNL (FN)	24 ± 4 [illegible] ± 4															
		NASA (D)	[illegible] [illegible] [illegible] [illegible]															
[illegible] [illegible]68	Lithium From charge pot and [illegible] after [illegible] Taken at 4[illegible]°F	GE (D)	[illegible]	[illegible]	[illegible]			[illegible]		5	<25	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]
		NRL							<25				[illegible]					
[illegible] [illegible]68	Lithium After [illegible] flush. Taken at 4[illegible]°F	GE (D)	[illegible]	19 5[illegible]	[illegible]			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				[illegible]
		NRL							<25				<25			[illegible]		[illegible]
		ORNL (FN)	[illegible] 41															

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TABLE K-I

CHEMICAL ANALYSIS OF THE ALKALI METALS FOR T-111 RANKINE SYSTEM CORROSION TEST LOOP

										Metallic Impurities, ppm in the Metal																	
B	Ba	Be	Ca	Cb	Co	Cr	Cu	Fe	Hf	K	Li	Mg	Mn	Mo	Na	Ni	Pb	Si	Sn	Sr	Ta	Ti	V	W	Zn	Zr	Remarks
		<5	133	<25	<5	<5	<5	<5				5	<5	<5	53	<5	<25	5	<25			<5	<25			<5	
		<5	53	<25	<5	<5	<5	<5				5	<5	<5	133	<5	<25	5	<25			<5	<25			<5	
<50		<5	5	<25	<5	<5	28	5				<5	<5	<5	80	28	<5	26	<25			<5	<5			<5	
15		50	300	ND	ND	100	15	200				2000	50	50	500	100	5	2000	ND	25		10	5			ND	
<50	<50	<5	<5	<25	<5	<5	<5	<5				<5	<5	<5	<25	<5	<25	<5	<25	<5		<25	<25			<25	
<50	<50	<5	<5	<5	<50	<5	<125	5				<5	<5	<5		<5	50	<5	<5	<5			<50			50	
<50	<25	<5	25	<25	5	<5	50	5				5	<5	<5	75	<25		<25	<25	5		<25	<25			<25	
<50	<50	<5	25	<25	<5	5	5	<5				5	<5	<5	125	<5	<50	<5	<50	5	<250	<5	<5			<25	
			<25			<25	<5	15		<25		10			150			<25			<250						

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Mn	[illegible]	Cb	[illegible]	Cr	Cu	Fe	[illegible]	K	Li	Mg	[illegible]	Mo	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	Remarks
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	125	[illegible]				[illegible]		[illegible]	[illegible]			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			[illegible]	
								≠500																[illegible]
	[illegible]	2	<2	<2	<2	2				<2	[illegible]	<2	[illegible]	[illegible]	<2	[illegible]			[illegible]	2			[illegible]	
<2	2	<10	<2	<2	[illegible]	[illegible]				<2	<2	<2	<20	<2	<10		<10		<2	<2			<20	
<2	<2	<10	<2	<2	<2	<2				2	<2	<2	2	<2	<2	<2	<2		<2	<10			<2	
[illegible]	[illegible]	<20	<2	500	<2	[illegible]			[illegible]	<2	2	<2	[illegible]	[illegible]	[illegible]	[illegible]	<2	<2	[illegible]	[illegible]			[illegible]	Lithium determined by flame photometry
<2	[illegible]	[illegible]	<2	<2	<2	<2				<2	<2	<2	<20	<2	<10	<2	<2	<2	<2	[illegible]			<10	
[illegible]	[illegible]	<20	<2	[illegible]	[illegible]	[illegible]				[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	<20	[illegible]	[illegible]	[illegible]			[illegible]	
<2	[illegible]	<10	[illegible]	2	<2	[illegible]				[illegible]	[illegible]	[illegible]		<10	<10	[illegible]	[illegible]	<2	[illegible]	<10	<100	<100	<10	
	<2	<20	<20	<5	[illegible]	[illegible]			[illegible]	1	<2	<2	[illegible]	<10	<5	[illegible]	<2		<2	<20				Lithium determined by emission spectrograph
<2	[illegible]	[illegible]	[illegible]	<2	[illegible]	[illegible]			[illegible]	<2	<2	<2	<20	<2	<10	[illegible]	<2	<2	<2	<20			<2	Lithium determined by flame photometry
<2	[illegible]	[illegible]	<2	<2	[illegible]	<2			[illegible]	<2	<2	<2	<2	[illegible]	[illegible]	[illegible]	<2	<2	<2	[illegible]			[illegible]	Lithium determined by flame photometry. A few black particles found at bottom of tube.
																								Lithium by flame photometry. Black particles found during preparation of chloride.
				Mi		Mi	ND			[illegible]	[illegible]			M		T		ND	Mi		ND	T	T	[illegible] amount of [illegible] lines very weak [illegible] minors, majors, traces, minors, and traces, not detected. Fe [illegible] 25% [illegible]; Mi [illegible] 50% [illegible]; [illegible] 5%; [illegible]; A [illegible] 5%, [illegible] 5%, [illegible] 05%. X-ray diffraction [illegible] and [illegible] Fe [illegible] Fe. X-ray diffraction [illegible] LiOH and Fe [illegible]
																								No [illegible] value
																								No [illegible] value
								[illegible]																Lithium [illegible] by flame photometry
									[illegible]															Lithium by flame photometry

FOLDOUT FRAME

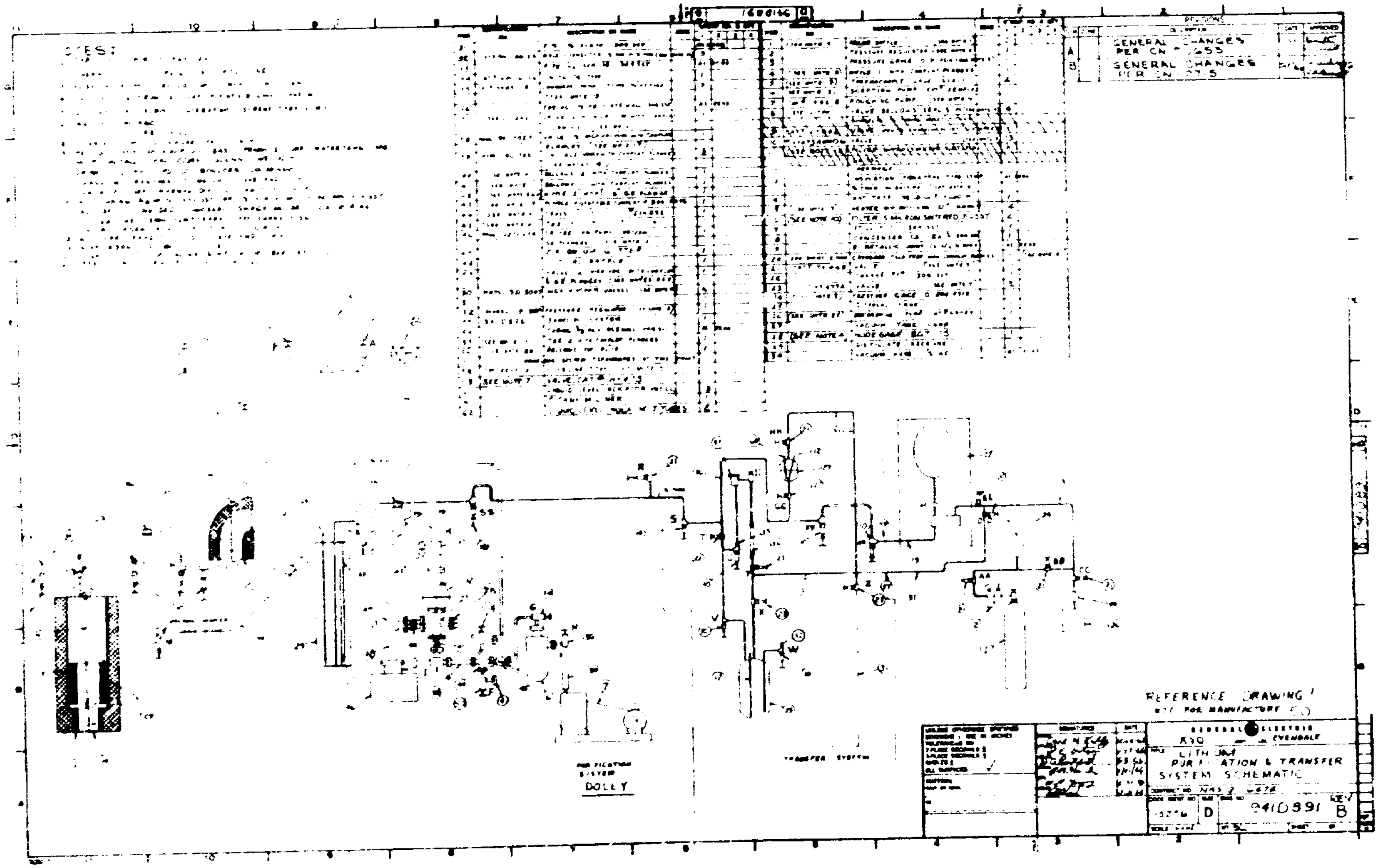


Figure K-2. Lithium Purification and Transfer System Schematic.

sample No. 1493B taken after flushing the loop. These results indicate that the leak between the primary and secondary circuits was not present during the initial loop filling and flushing operations.

Potassium sample No. 1668A, removed from the bottom third of the sample tube shown in Figure K-1, which was obtained during the draining of the potassium loop, was also analyzed for lithium by flame photometry. The analysis indicated 8 ppm lithium. Again, all other metallic impurities were at or below the detection limits. This sample tube was kept above the potassium melting point overnight after the draining operation to allow any particulate matter present to settle to the bottom. The end cap was removed with a tubing cutter and the potassium dissolved in alcohol.

Potassium sample No. 1668B was removed from a portion of the heavy-walled tubing used as the drain line between valve KK and the still pot (Figure K-1). A flame photometric analysis of this sample indicated 3.3 percent lithium. Some black particulate matter was found in the flush water and was analyzed spectrographically (sample No. 1668C). This qualitative analysis indicated minor quantities of Cr, Fe, Ni, and Ti and trace quantities of Al, Ca, Mg, Mn, Si, Zn, and Zr, but no Ta, Hf, or W were detected.

Sample No. 1669A consisted of the black particulate residue which remained in the potassium still pot after distillation. The results of the semiquantitative emission spectrographic analysis performed by National Spectrographic Laboratory indicated 25-50% Cr, 25-50% Mn, 15-25% Fe, 5-15% Ni, 5-15% Si, 0.1-0.5% Al, 0.1-0.5% V, and < 0.05% Ta.

Two specimens of the black residue were prepared for X-ray diffraction analysis. The first (sample No. 1669B1) was placed in the glass capillary in an argon-filled glove box, while the second (sample No. 1669B2) was loaded in air. A best-fit analysis of the diffraction data indicated the presence of β-$LiFeO_2$ and α-$LiFe_5O_8$. However, after standing in air for a few days, another diffraction pattern was obtained which again indicated the presence of lithium iron oxides, as well as Li_2O and α-Fe. The diffraction pattern of the second specimen indicated the presence of lithium, Li_2O, LiOH, and α-Fe. No Cr, Mn, or Ti was noted for either sample.

C-5

A sample (No. 1672) of the metal, remaining in the still pot after distillation of the potassium, was removed by dissolving in water. The results of flame photometric analyses indicated approximately 100 percent lithium and 600 ppm potassium. A flame photometric analysis of the distilled potassium (sample No. 1682) indicated 20 ppm lithium.

The conclusions drawn from the operations and analyses just presented were as follows:

1. A leak existed in the boiler of the loop between the primary and secondary circuits;
2. The plugging of the metering valve was due to particulate matter which probably originated from stainless steel;
3. The chemical analytical results did not provide a clear-cut answer to when the leak first appeared, but indications are that it occurred after the loop was filled with the alkali metals;
4. The chemical analytical results did not provide a clear-cut answer as to the source of the particulate matter although the indication is that it was introduced from the potassium transfer lines and charge pot.

The loop was prepared for draining of the lithium circuit and distillation of the residual alkali metal using procedures established previously with the Cb-1Zr Rankine System Corrosion Test Loop.[35] Six quartz lamps[a] with tungsten filaments were installed in stainless steel brackets welded to the loop support structure. A stainless steel thermal shield was wrapped around the loop support structure to thermally insulate the loop and quartz heaters from the test chamber bell jar. The lithium and potassium surge tanks were partially shielded from the quartz heaters

(35) Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, NASA CR-1509, June 1970.

(a) General Electric Type 3800 Te/VB, 3800 watts, 570 volts, 1/2 inch OD x 43 1/2 inches long.

to maintain a lower temperature than the loop so that the alkali metal distilled from the loop would condense in the cooler surge tank located in the lower portion of the vacuum chamber.

The potassium still, used in the initial drain of the secondary circuit previously described, was reassembled and reattached to valve KK of the potassium circuit, omitting the valve between valve KK and the still pot.

The lithium drain container, shown in Figure K-3, was fabricated and attached to valve KK of the primary circuit. This container was fabricated from a 24-inch (61-cm) section of 4-inch, schedule 10, Type 304 stainless steel pipe and 4-inch, Type 304 stainless steel pipe caps, and had a volume of 6600 cc. It was weighed prior to attachment to the drain line and the vacuum-argon line.

Both the lithium and the potassium drain systems were baked out and helium leak-checked per NSP Specification No. 03-0013-00-B, and no leaks were found in excess of 5×10^{-10} std cc of air per second at temperatures from 400° to 700°F (204° to 371°C). The potassium circuit had previously been drained of liquid metal.

After a low-power electrical checkout of the heating lamps, the bell jar was lowered to the spool section, and the vacuum chamber was sealed. The turbomolecular pump was used to rough-pump the vacuum chamber for 24 hours before the ion pump was turned on. The chamber bakeout was turned on when the chamber pressure was in the 10^{-7} torr (10^{-5} N/m^2) range. The system was outgassed for approximately 100 hours with only the vacuum system bakeout on. The loop temperature during this period was approximately 500°F (260°C). The EM pump power was turned on at approximately 10 percent of the rated power to heat up the pump duct.

The lithium EM pump power was increased to 40 percent of rated power and the lithium loop temperature increased to 1000°F (538°C). After circulating lithium for 4 hours, the lithium circuit was evacuated and the lithium drained from the loop. After draining the lithium circuit, the EM pump power was reduced to 10 percent of the rated power to maintain the pump duct temperature at 1000°F (538°C).

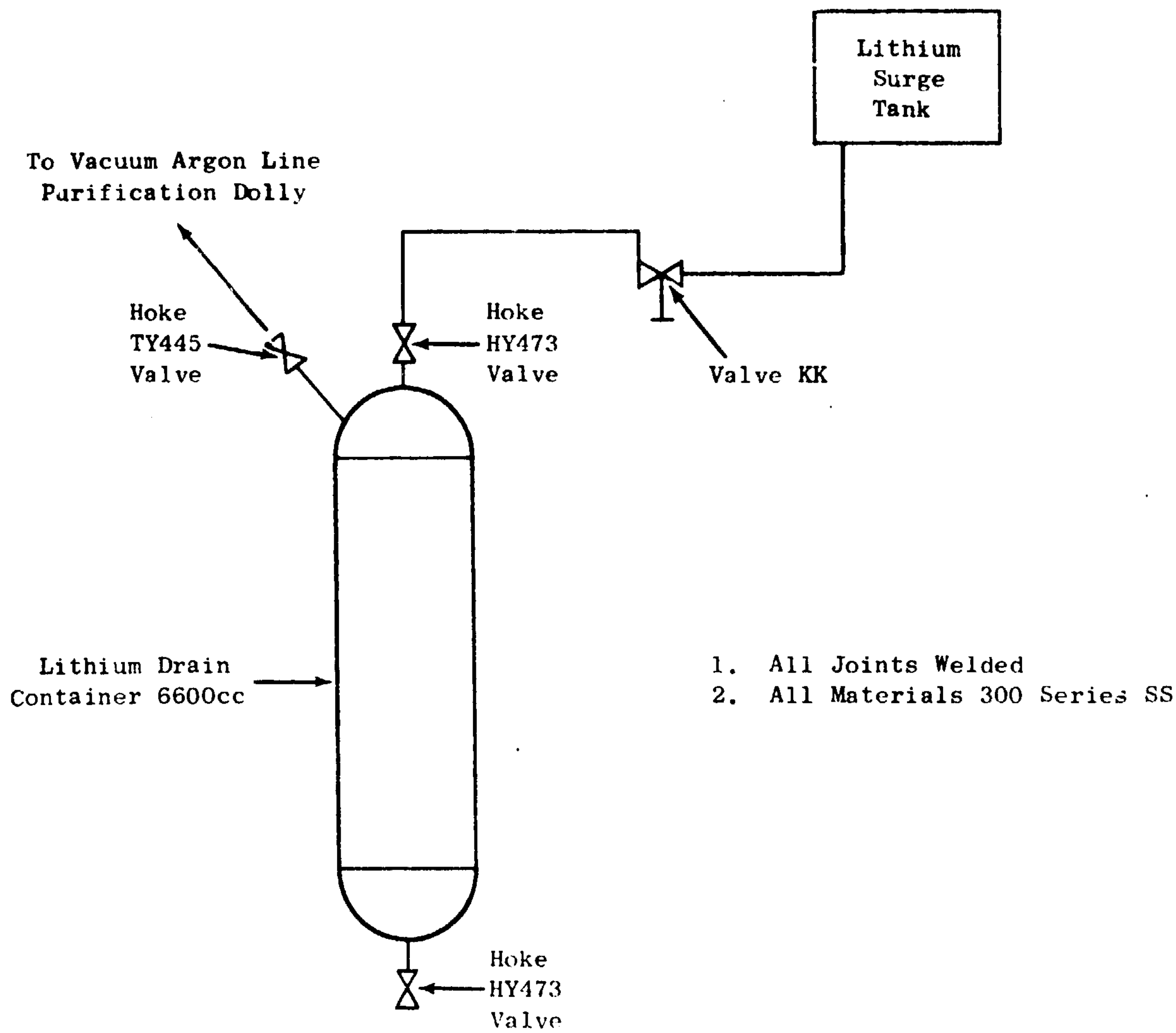


Figure K-3. Schematic of System Used for Draining Lithium From the Primary Circuit of the Loop.

Approximately 3600 cc of metal was removed from this circuit as indicated by level probe measurements of the liquid level in the container. The level probe also indicated an additional interface about 1 1/2 inches (3.8 cm) above the bottom of the container which corresponded to about 200 cc of potassium. After cooling the container to room temperature, a radiograph showed the interface of the two layers, thus confirming the presence of a substantial quantity of potassium in the lithium circuit of the loop.

After the initial draining operation, both circuits were evacuated with the vacuum system on the potassium purification dolly. The quartz lamp power was then slowly increased in small increments to hold the vacuum chamber pressure in the 10^{-7} torr (10^{-5} N/m^2) range. The potassium EM pump power was increased, and the power to the potassium preheater was turned on.

Typical operating conditions during the ten-day distillation period are as follows:

Chamber Pressure	3×10^{-8} torr (4×10^{-6} N/m^2)
Average Lithium Circuit Temperature	750°F (399°C)
Average Potassium Circuit Temperature	750°F (399°C)
Boiler Temperature	850°F (454°C)
Potassium Preheater Temperature	1300°F (704°C)
Lithium Surge Tank	540°F (282°C)
Potassium Surge Tank	580°F (304°C)
EM Pump Duct Temperature (Li and K)	1200°F (649°C)

During the distillation period, the vacuum chamber bakeout heaters on the spool section and the sump were not turned on so that the lower portion of the loop (surge tanks) would be at a lower temperature to condense the alkali metal, distilled from the loop, in the surge tanks.

Periodically, the surge tank drain valves were opened to gravity-drain the distillate from the surge tank. The surge tank would then be pressurized to drive any remaining alkali metal into the drain tank.

3. Preliminary Leak Checking of the Boiler Before Cutting

The methods to be employed in repairing the boiler were contingent upon the location of the leak between the potassium and lithium circuits.

Some effort was thus expended in localizing the leak before the boiler was removed from the loop.

An attempt to locate the leak was made by measuring the steady-state leak rate as determined by a helium mass spectrometer leak detector. The lithium loop was pressurized with helium to 0.4 psia. The potassium circuit was evacuated to the 10^{-4} torr (10^{-2} N/m^2) range with the helium leak detector. At these conditions a steady-state reading on the helium level meter of the leak detector could be stabilized at 7.5 x 10^{-8} std cc/sec of helium. Any change in the helium leak rate would be indicated by a corresponding change on the helium level meter. The inlet to the boiler was then heated to 380°F (193°C) for approximately 20 minutes with a slight decrease in the helium level as shown in Figure K-4. The heater power to the boiler inlet was turned off, and the boiler outlet was heated to 400°F (204°C). Again, the helium level decreased at the same rate as observed when the inlet was heated. When the center section of the boiler, which consists of seven 10-inch (25.4-cm)-diameter coils, was heated, the helium level increased indicating a higher leak rate. The leak rate continued to increase even after the power was turned off. Subsequently, with the boiler at room temperature, the lithium circuit was pressurized with argon to 50 psia (3.45 x 10^5 N/m^2) while the potassium circuit was evacuated. The boiler remained at these conditions for three days without indicating a leak as evidenced by the lack of a drop in pressure in the lithium circuit.

The results of these tests indicated that the leak might be in the center section of the boiler and that the leak was only apparent when the boiler was heated above the melting point of lithium (367°F, 180.6°C).

3. Boiler Cutting Procedures

The procedures employed in cutting the boiler were such to minimize the possibility of contaminating the loop either by air reacting with the residual alkali metal in the loop once a cut was made or by chips from the cutting operation falling into the loop. To prevent contamination of the alkali metal, all cutting operations were performed in inert gas (argon) atmosphere. To accomplish this, a vinyl, 0.020-inch (0.51-mm)-thick chamber was designed, fabricated, and assembled around the loop.

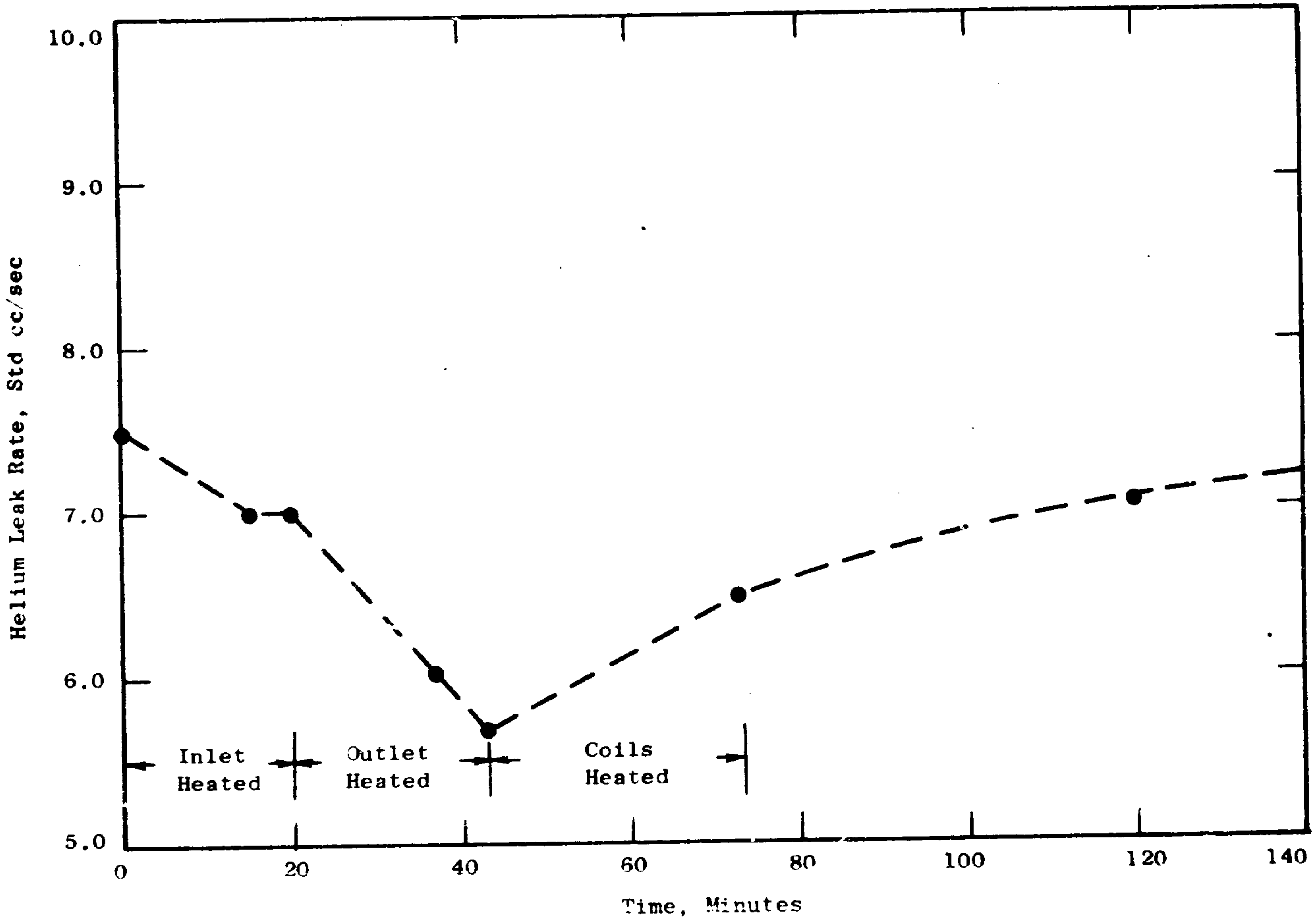


Figure K-4. Effect of Heating Localized Sections of the T-111 Boiler on Helium Level in Evacuated Potassium Circuit Measured With a General Electric M-60 Helium Leak Detector.

The chamber shown in Figure K-5 is fitted with neoprene gloves appropriately located for access to the areas to be cut at the top and bottom of the boiler. The chamber was sealed from the atmosphere at its base by a rubber-cushioned stainless steel expansion ring pressing the vinyl against the inside of the vacuum chamber spool piece. The vacuum bell jar could therefore be lowered over the vinyl chamber and mated with the spool piece flange without interference at the sealed end of the vinyl chamber. This arrangement provided the capability of evacuating the vinyl chamber inside the bell jar. The annulus between the vinyl chamber and the bell was also evacuated to prevent collapse of the chamber. Following an overnight pump-down, the chamber was backfilled with argon. Analysis of the exiting argon was performed with a Beckman Model 80 Trace Oxygen Analyzer and a Model 27901 electrolytic hygrometer shown in Figure K-6. Typical analyses of less than 10 ppm oxygen and less than 70 ppm water vapor were obtained. Subsequently, the bell jar would be removed and cutting operations would be initiated. Gas analysis was continued during operations in the vinyl chamber. The water vapor concentration in the chamber increased with time but typical analyses indicated maximum concentrations of less than 200 ppm when the gas-filled chamber was maintained at ambient conditions. Only small increases in oxygen concentration were noted.

All boiler cuts were made with a tubing cutter to eliminate chip formation which could fall into the loop. The cutters were fitted with three cutting wheels such that cuts could be made without 360^{o} rotation.

4. Removal of the Boiler From the Loop

The welds separating the potassium and lithium circuits of the boiler were thought to be the most probable locations for the leakage to occur. The locations of these welds are shown on the sketch of Figure K-7. If the leak had been in the weld at the top of the boiler, the boiler could have been repaired without removal from the loop. The initial cut was therefore made to expose this weld for inspection and leak checking and subsequent repair if required.

The boiler was cut exposing the top weld and the potassium circuit, which had been under a positive argon pressure, was immediately sealed with expandable plugs (Imperial Model 140F Tubing Test Plugs) as shown in Figure K-8. The seal is accomplished by a rubber grommet expanded

Figure K-5. Vinyl Chamber Used for Cutting Operations on the Boiler.
(Orig. C68062842)

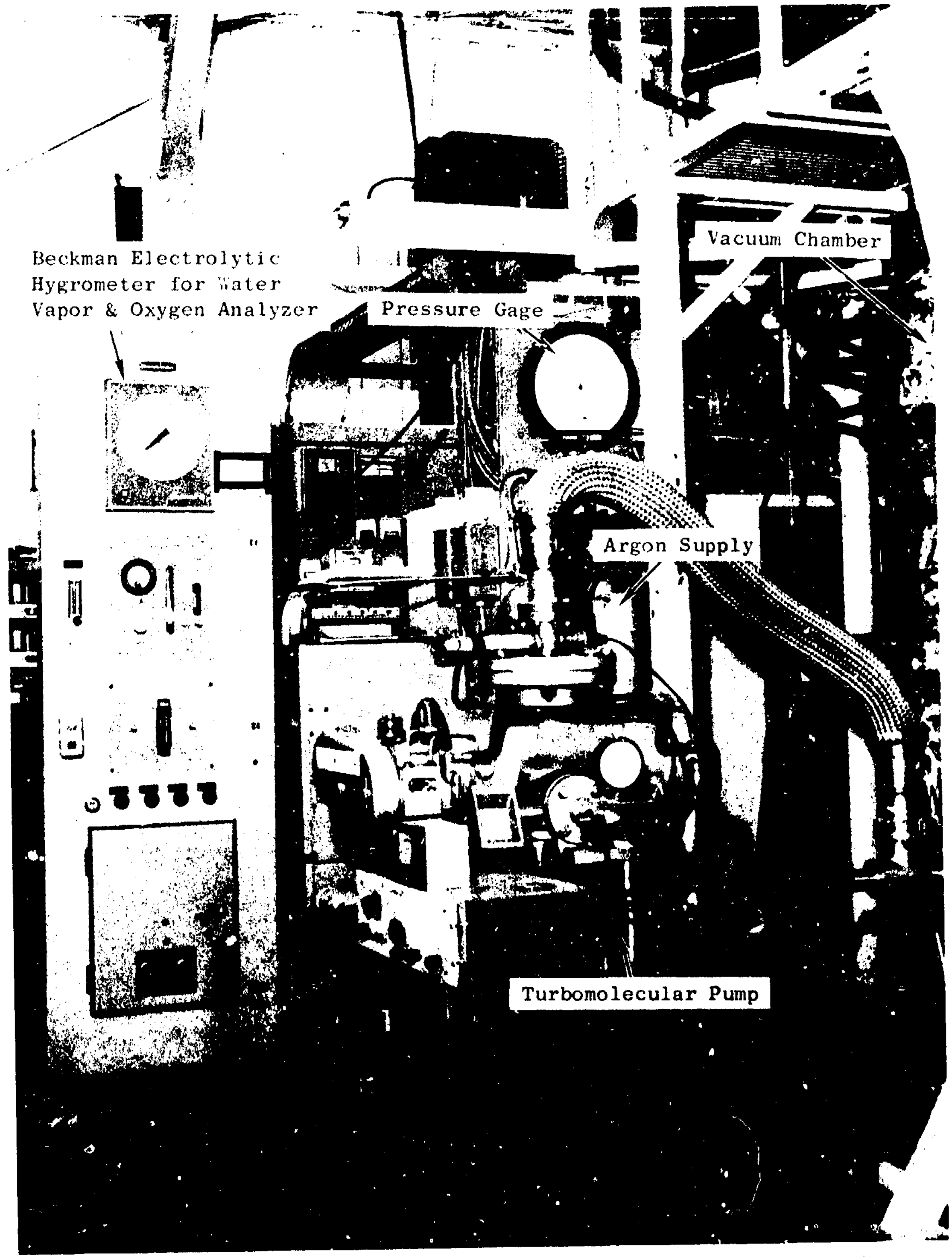


Figure K-6. Vacuum Equipment and Instrumentation Used for Evacuation and Backfilling of Vinyl Chamber and Analysis of Exiting Inert Gas. (C68062846)

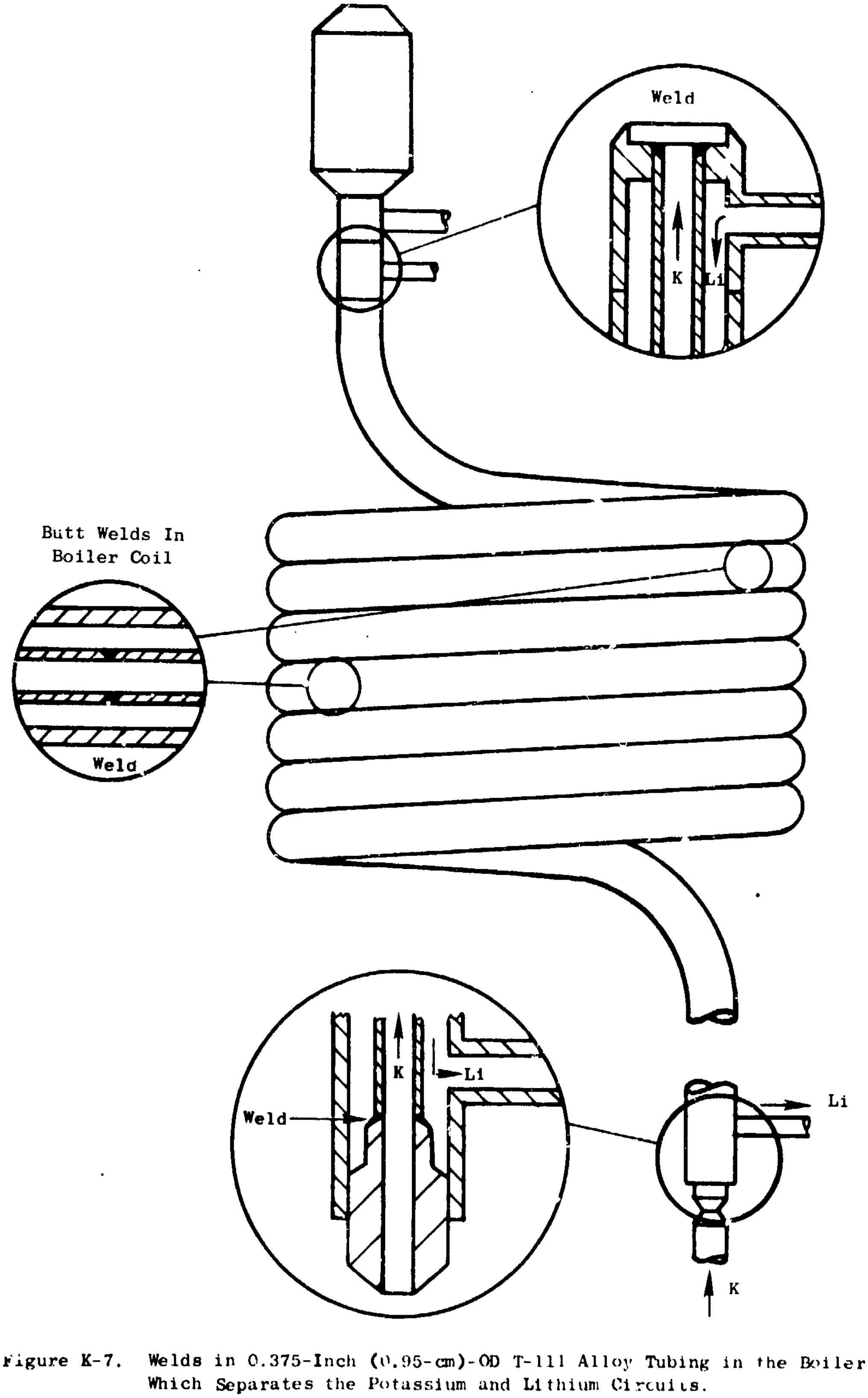


Figure K-7. Welds in 0.375-Inch (0.95-cm)-OD T-111 Alloy Tubing in the Boiler Which Separates the Potassium and Lithium Circuits.

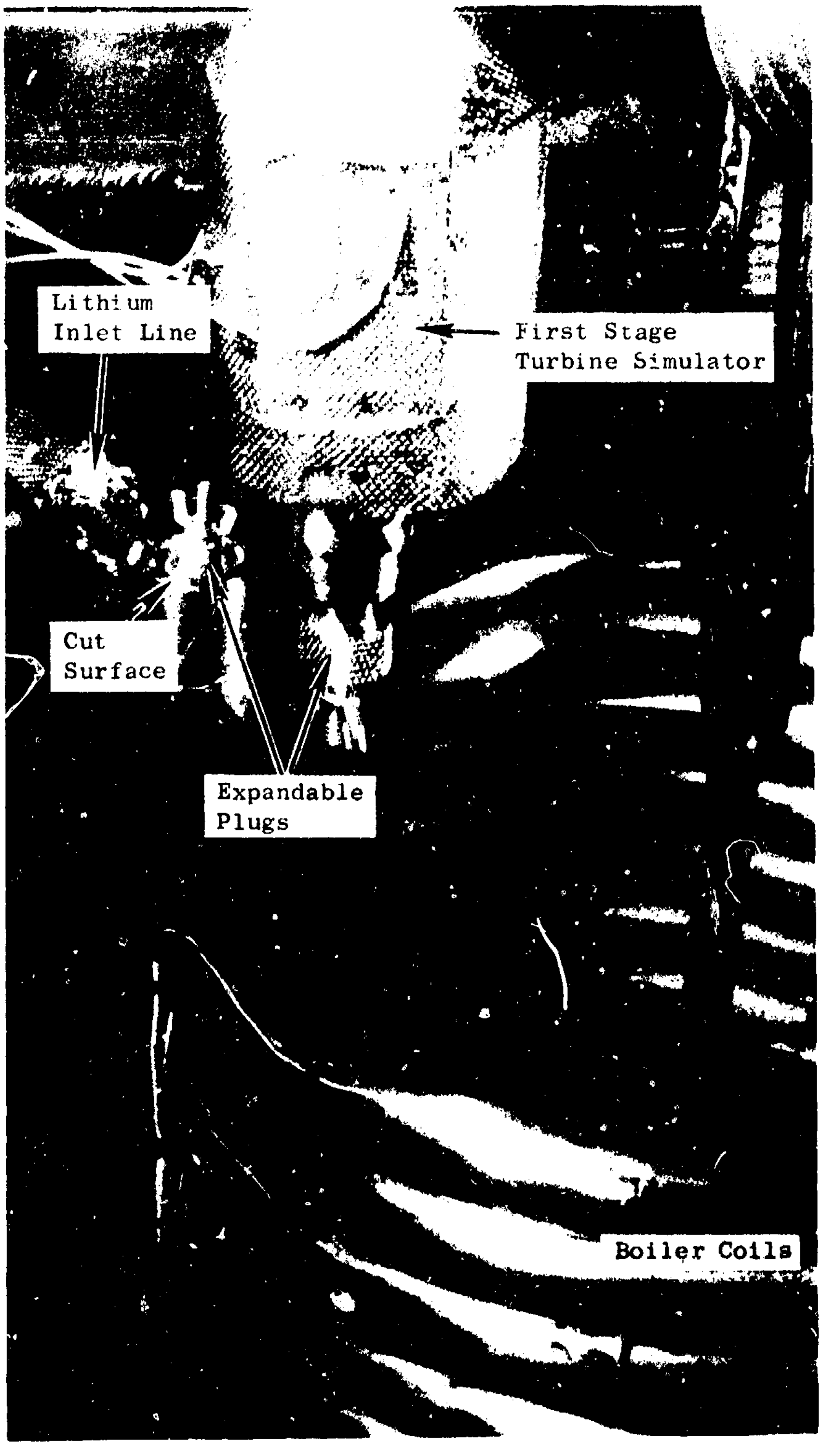


Figure K-8. T-111 Boiler Following Initial Cut. The Potassium Circuit Was Sealed From Atmosphere with Expandable Plugs. (Orig. C68062845) against the inside diameter of the tube by the action of a brass sleeve actuated with the wing nut as shown in Figure K-9.

Inspection and helium mass spectrometer leak checking of the weld did not reveal a leak at this location. Subsequent room temperature leak checking of the boiler coils and the bottom of the boiler also did not reveal a leak. The vinyl chamber was removed.

The boiler was heated to 400°F (204°C) at various locations, as previously described, with helium in the potassium circuit and the mass spectrometer leak detection pumping on the lithium circuit. Again, no leaks were found at the top of the boiler; however, definite leak indications were noted when the center coils were heated to 400°F (204°C). The leak could not be detected when the boiler was returned to room temperature. Similar behavior was obtained in the previous leak tests which suggests that the leak could be a fine crack which could plug the residual lithium (M.P. 357°F, 180.6°C).

Since it could definitely be determined that the leak was not at the top weld, and this was the only weld that could be repaired in place, preparations were made to cut the boiler from the loop. Three additional cuts were required to remove the boiler; the lithium inlet line, the lithium exit line, and the potassium inlet just above the top electrode as shown in Figure K-10. The vinyl chamber was reinstalled, and the lithium inlet and outlet lines were cut and sealed. Similar techniques were employed as previously described, with the exception of the seals on the boiler which were made with Swagelok fittings as shown in Figure K-11. The Swagelok caps were substituted for the expandable plugs to facilitate subsequent attachment of the boiler to the cleaning apparatus for the removal of residual alkali metal. The final cut to remove the boiler was made at the potassium inlet. The wall thickness was reduced by hand filing to 0.125 inch (0.32 cm) over a 0.750-inch (1.91-cm) length and V-grooved in the area to be cut to 0.050 inch (0.13 cm) prior to final cutting with the tubing cutter within the vinyl chamber.

5. <u>Cleaning the Boiler</u>

The proposed method of removing the residual potassium and lithium from the T-111 boiler by flushing with anhydrous liquid ammonia (99.999%,

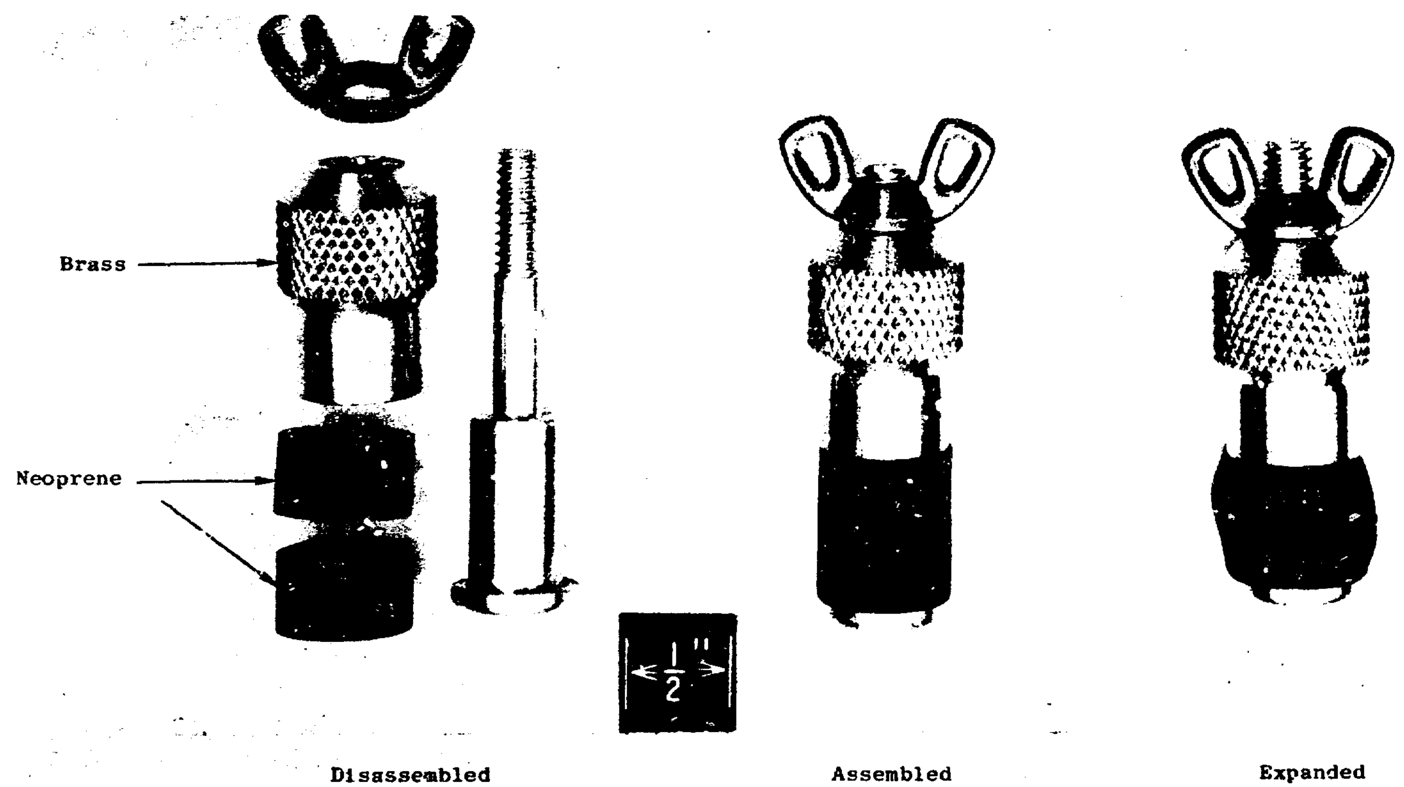


Figure K-9. Expandable Plugs Used to Seal the Loop Tubing After Cutting. (Orig. C680719136)

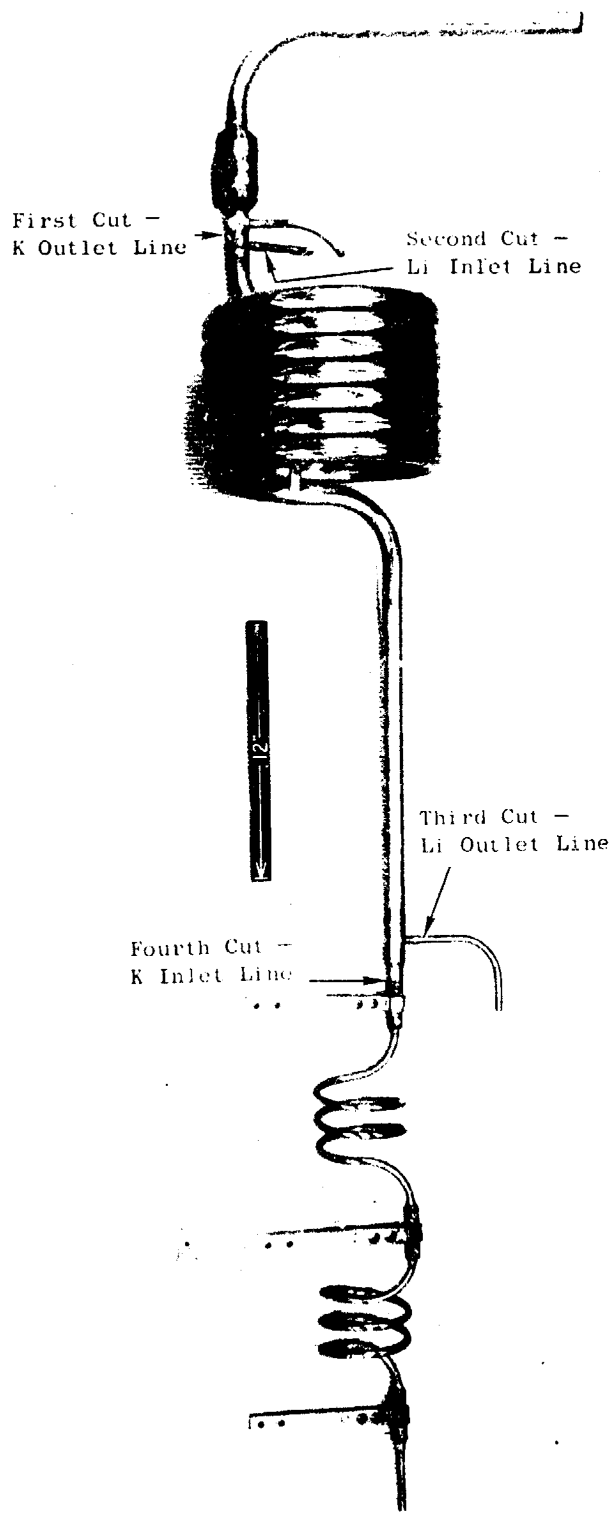


Figure K-10. Location of Tubing Cuts Made to Remove Boiler From Loop as Indicated on the Boiler Assembly Before Final Loop Assembly. (Orig. C67100412)

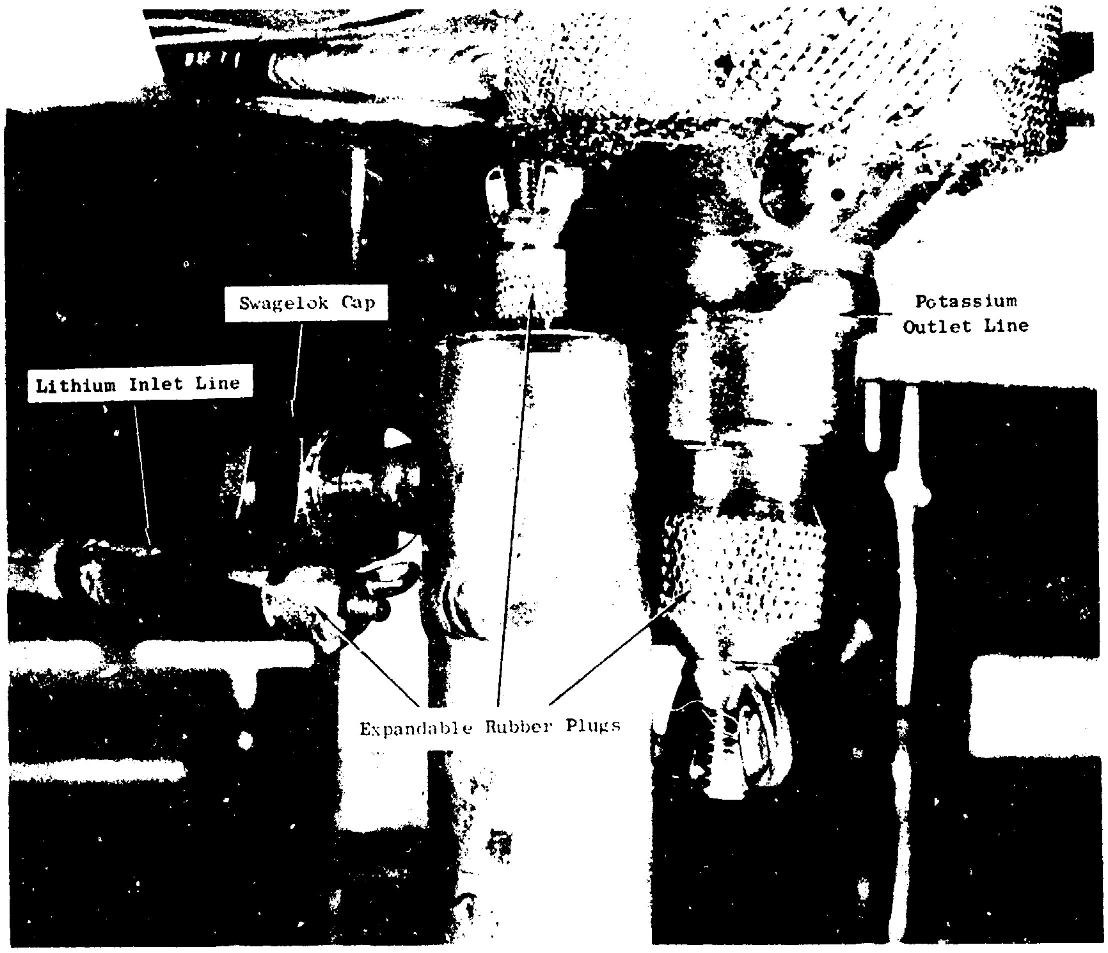


Figure K-11. Top of Boiler After Cutting and Sealing Lithium Inlet and Potassium Exit Lines. (C680719137)

< 10 ppm H_2O) was tested by cleaning a simulated boiler constructed of 0.5-inch (1.27-cm)-OD, type 316 stainless steel tubing. This apparatus consisted of two coils and contained a section of 1/8-inch (0.32-cm), stainless steel rod around which were wrapped specimens of 0.060-inch (0.15-cm)-diameter T-111 and Cb-1Zr wire. This special section was included to simulate the plug region of the T-111 loop. The lithium drain container, containing the lithium and a small amount of potassium removed from the loop, was emptied into another container through the simulated boiler. Argon was then blown through the apparatus so that most of the alkali metal was removed. The residual alkali metal was then flushed out with liquid ammonia using the procedure described below for the actual boiler. The T-111 and Cb-1Zr specimens showed no significant change in carbon, hydrogen, oxygen, or nitrogen concentrations as a result of the cleaning operation, thus qualifying the method for cleaning the T-111 boiler.

The T-111 boiler, previously sealed in an argon atmosphere, was connected to the liquid ammonia system as shown in Figure K-12, and the system was evacuated up to the liquid ammonia valve. The system was back-filled with argon, and the lithium and potassium circuits were tested individually to determine if a gas passage existed in each circuit. The lithium circuit was subsequently capped, and the argon flow was adjusted to 50 ml/min through the potassium circuit. With this argon flow maintained, the boiler and liquid ammonia cooling coil were cooled to $-50^{\circ}C$ by pouring liquid nitrogen (B.P. $-196^{\circ}C$) into a 50-percent-by-volume mixture of methanol and water. When the boiler reached $-50^{\circ}C$, the temperature necessary to maintain the ammonia in a liquid state, the argon flow valve was closed as the valve to the ammonia bottle was opened initiating ammonia flow. The first portion of liquid ammonia emerging from the boiler was dark blue in color as a result of the ammonia-potassium interaction, but after 500 ml of liquid ammonia had passed through the boiler, the effluent was clear, indicating that the bulk of the potassium had been flushed out in about 10 minutes. The flushing was continued for an additional 45 minutes, at which time about 2000 ml of liquid ammonia had passed through the boiler.

The procedure for cleaning the potassium circuit was applied to cleaning the lithium circuit with similar results. The lithium circuit contained

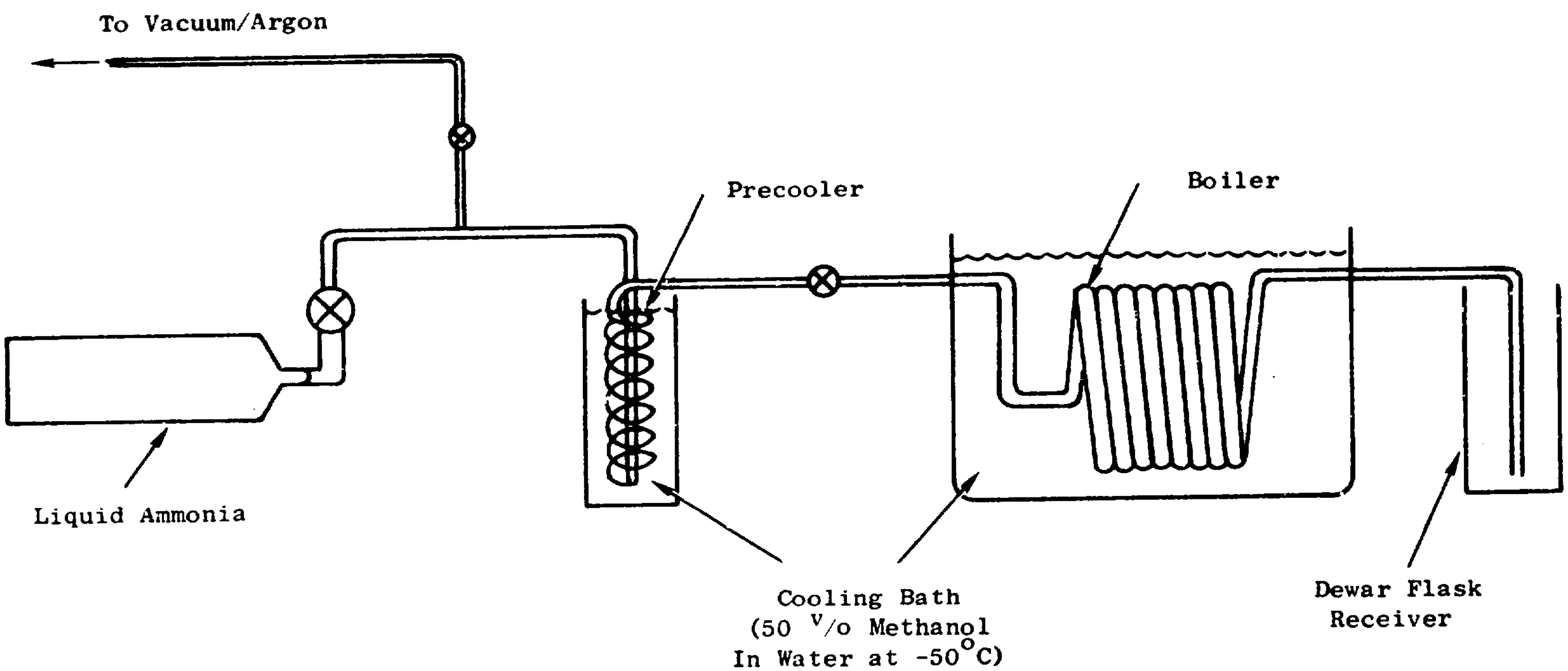


Figure K-12. Schematic of Apparatus Used to Removed Residual Alkali Metal From Boiler by Dissolution in Liquid Ammonia.

a greater quantity of alkali metal than the potassium circuit and required a greater quantity of liquid ammonia to remove the bulk of the lithium. The liquid ammonia flushing of the lithium circuit was also continued for 45 minutes after the disappearance of the blue color.

Both circuits were purged with argon at room temperature to remove residual ammonia. They were then filled with absolute ethanol which was drained after a 48-hour soak time in the boiler. The circuits were again filled with ethanol which was subsequently analyzed by flame photometric techniques. No lithium or potassium was indicated in the analysis (< 10 ppm Li, < 10 ppm K). This procedure was repeated using distilled water with similar analytical results. The boiler was finally dried with an ethanol rinse and argon purge in preparation for locating the leak.

6. Locating the Leak in the Boiler

The cleaned and dried boiler was subsequently attached to a General Electric M-60 helium mass spectrometer leak detector. With the leak detector pumping on the lithium circuit, a helium flow was established through the potassium circuit. A leak was observed with a quantitative leak rate of 8×10^{-5} std cc/sec equivalent air leakage.

The general location of the leak was determined by use of ethanol and a leveling bulb attached to the potassium circuit. The boiler was placed in an upright position with the potassium exit line at the top, and a 250-cc leveling bulb half filled with ethanol was attached to the potassium inlet line with rubber tubing. The leveling bulb was adjusted so that the level of the ethanol within the potassium tube was near the bottom of the boiler. A flow of helium was maintained into the potassium exit line to keep helium in the vapor spaces above the ethanol level, and the leak detector was adjusted to give a high output reading. By slowly raising the leveling bulb, the liquid level within the boiler was moved upward until the ethanol covered the leak as indicated by a sudden decrease in the reading of the output meter. Using this procedure, the leak was located within the coiled section of the boiler, in the second turn from the top.

The ethanol was then drained from the boiler and the helium flow continued until the boiler was dry and the leak reappeared. The boiler was then inverted (potassium inlet line at the top), and the same procedure was repeated. The indicated leak location was at approximately the same place in the boiler as in the previous test. However, since the exact shape of the liquid interface within the boiler was not known, the precise position of the leak could not be determined. At this point, it was estimated that the leak was known to be within a section of the boiler tube approximately 20 inches long.

The ethanol was again drained from the boiler and the boiler dried until the leakage was reestablished. The boiler was then placed in a horizontal position, and the leveling bulb was positioned level with the axis of the coil. The boiler was then slowly rotated about its axis so that the liquid interface within the boiler moved along the length of the tube. Using this technique, the most probable leak location was narrowed to a section of tubing approximately 6 inches long.

Inspection of radiographs of the boiler, which had been previously obtained, indicated that one of the butt welds in the potassium tube was near the center of the section of tubing in which the leak had been localized. It was concluded at this time that in all probability the leak was in this weld.

The boiler was then cut apart, and the suspected section of potassium tube containing the butt weld was removed. The section was leak-checked, and no leakage was found at first. However, after soaking the tubing in boiling water for 15 minutes, the leak reappeared and was localized using a fine helium jet. Using this helium probe technique, the leak was found to be near the center of the weld and within an area about 0.1 inch (0.25 cm) in diameter. Very careful inspection of this area with a microscope revealed some surface irregularities but not obvious cracks or pinholes.

The tube was pressurized with air, and the suspected leak area was coated with a soap film and observed at 30X magnification. Bubbles were observed at the leak as shown in Figure K-13. Close examination indicated a grain-boundary crack as the source of the leak which is shown in Figure K-14.

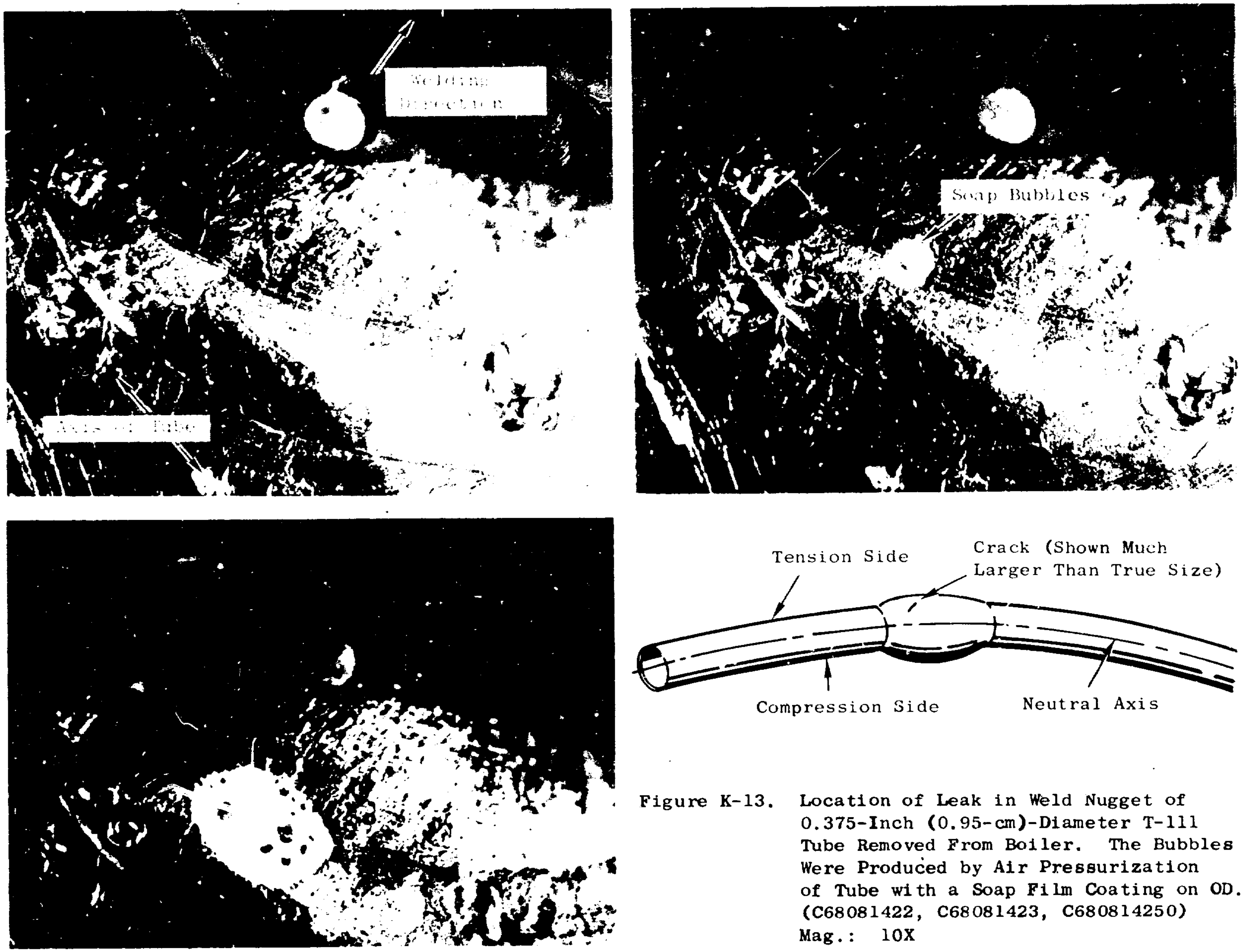
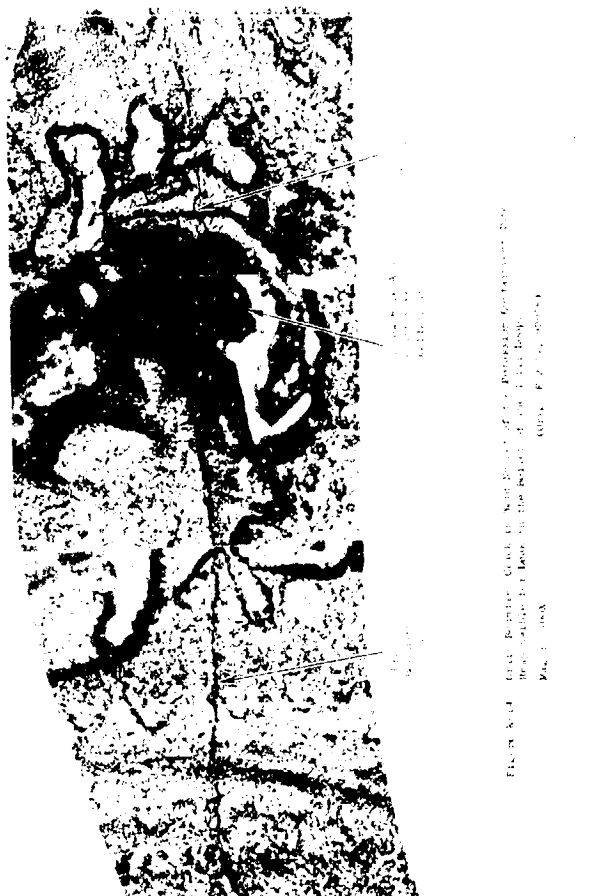


Figure K-13. Location of Leak in Weld Nugget of 0.375-Inch (0.95-cm)-Diameter T-111 Tube Removed From Boiler. The Bubbles Were Produced by Air Pressurization of Tube with a Soap Film Coating on OD. (C68081422, C68081423, C680814250) Mag.: 10X



Subsequently, the weld nugget section was removed from the tube and ground longitudinally up to the crack area in preparation for metallographic examination. At this time, a similar but much shorter crack was observed at 30X magnification on the ID of the tube.

7. Metallographic Examination of the Crack in the T-111 Alloy Boiler Tube

The weld nugget section of the tube butt weld containing the crack was removed from the boiler and ground longitudinally up to the crack area in preparation for metallographic examination. The specimen was mounted in clear epoxy such that the tube could be examined longitudinally, transverse to the crack. The specimen was Automet-ground* on 600-grit paper up to the fiducial marks which were placed on the specimen before mounting to indicate the location of the crack. Final polishing was accomplished using standard procedures.

The crack was observed to be intergranular and, as shown in Figure K-15, View AA, did not extend completely from the OD to the ID of the weld nugget. The specimen was subsequently repolished removing only approximately 0.002 inch (0.05 mm) from the surface. The appearance of the crack was similar as shown in View BB of Figure K-15 but shorter in depth. An additional polish was performed removing 0.006 inch (0.15 mm) of material from the surface. The crack appearance after this polish was considerably different and not as deep as observed previously, as can be seen in View CC of Figure K-15. It became clear at this point that the orientation of the specimen in the mount was such that the egress of the crack at the ID had been passed in the initial polishing step. In any event, examination of the crack at higher magnifications, performed between the polishing steps previously described, did sufficiently indicate the morphology of the crack.

The crack deviates from a simple shortest-path route, and entire grains are delineated by the grain-boundary separation, as can be seen in Figure K-16. The appearance of grain-boundary separation suggests some action of the lithium which has penetrated into the crack. The intermittent

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* Automatic Metallographic Polishing Apparatus - Buehler Ltd., Evanston, Illinois.

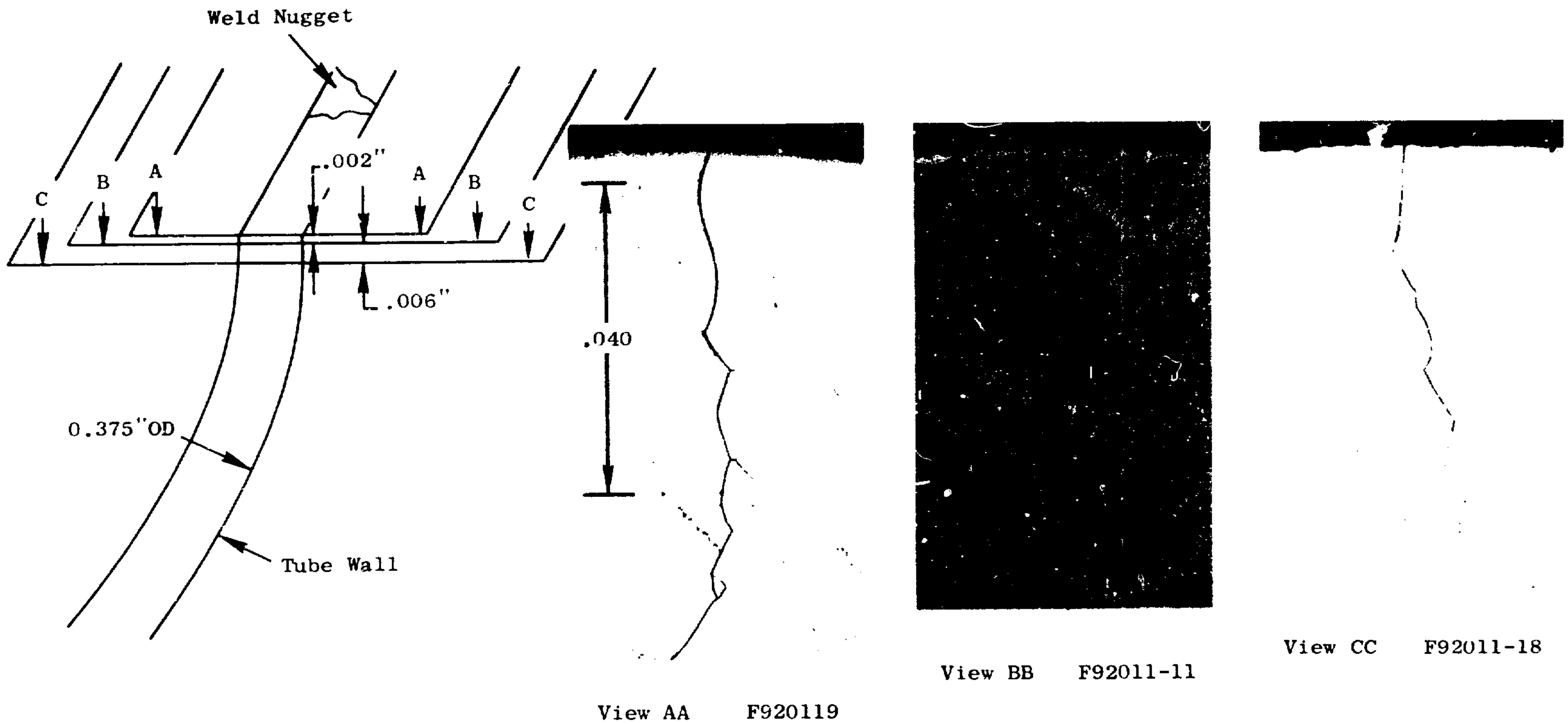


Figure K-15. Intergranular Crack in the Weld Nugget of the Potassium Boiler Tube Removed From the T-111 Loop.

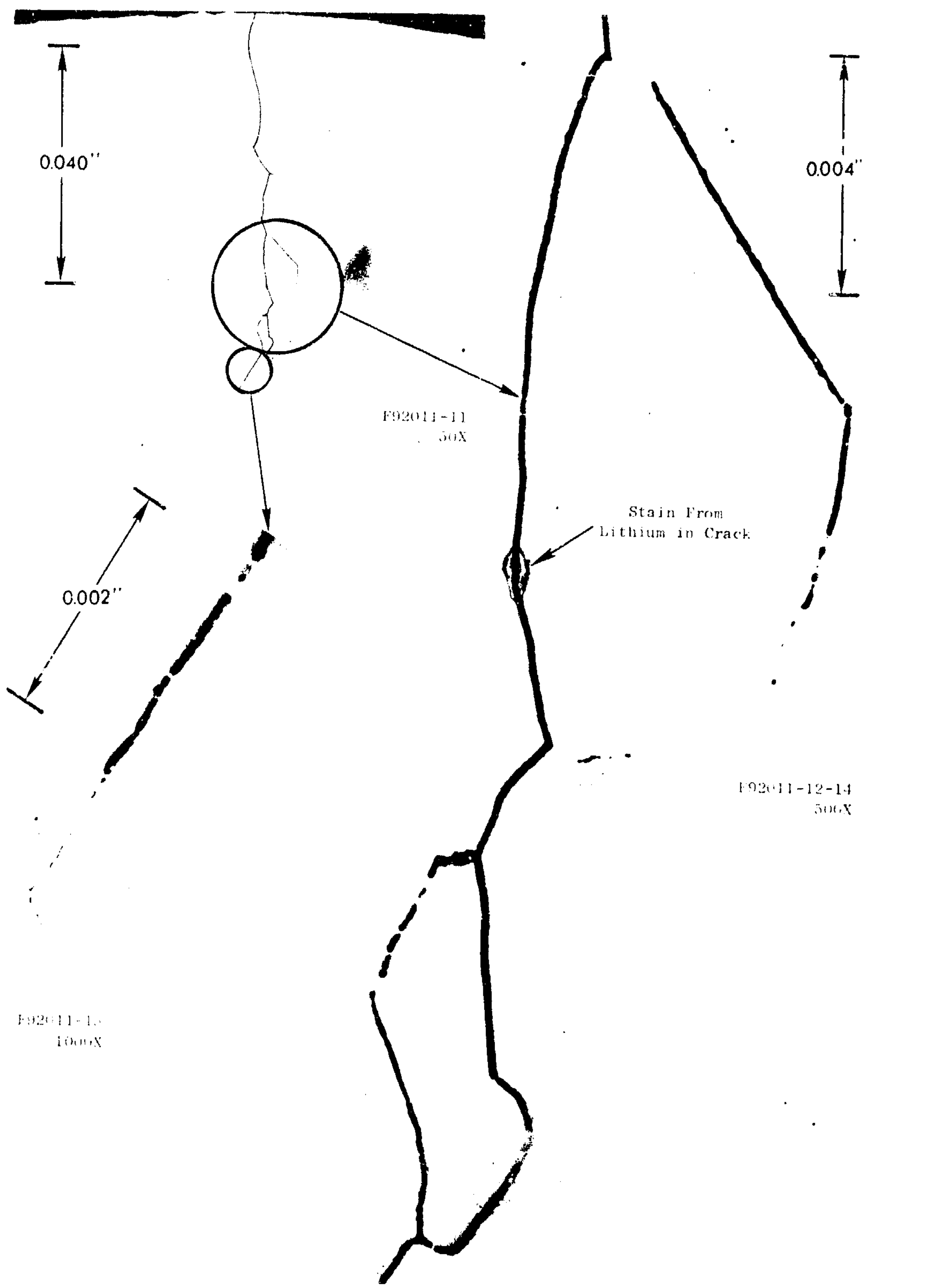


Figure K-16. Intergranular Crack in the Weld Nugget of the Boiler Tube.

gaping of the grain boundaries, especially off from the main crack and at the bottom of the crack, as shown in Figure K-16, is similar in appearance to lithium corrosion. A mechanism of attack of the weld by lithium is difficult to understand since no corrosion by lithium has been observed in T-111 weldments having such a low oxygen concentration (40 ppm). The role of lithium in the resulting metallographic structure is further exemplified in the microstructure at the bottom of the crack as shown in Figure K-17. The grain-boundary voids illustrated are difficult to explain in terms of cracking alone. The microstructures shown in Figures K-16 and K-17 are of areas in the specimen below the main crack path as indicated by their appearance as well as the fact the crack depth is decreasing with each additional polish.

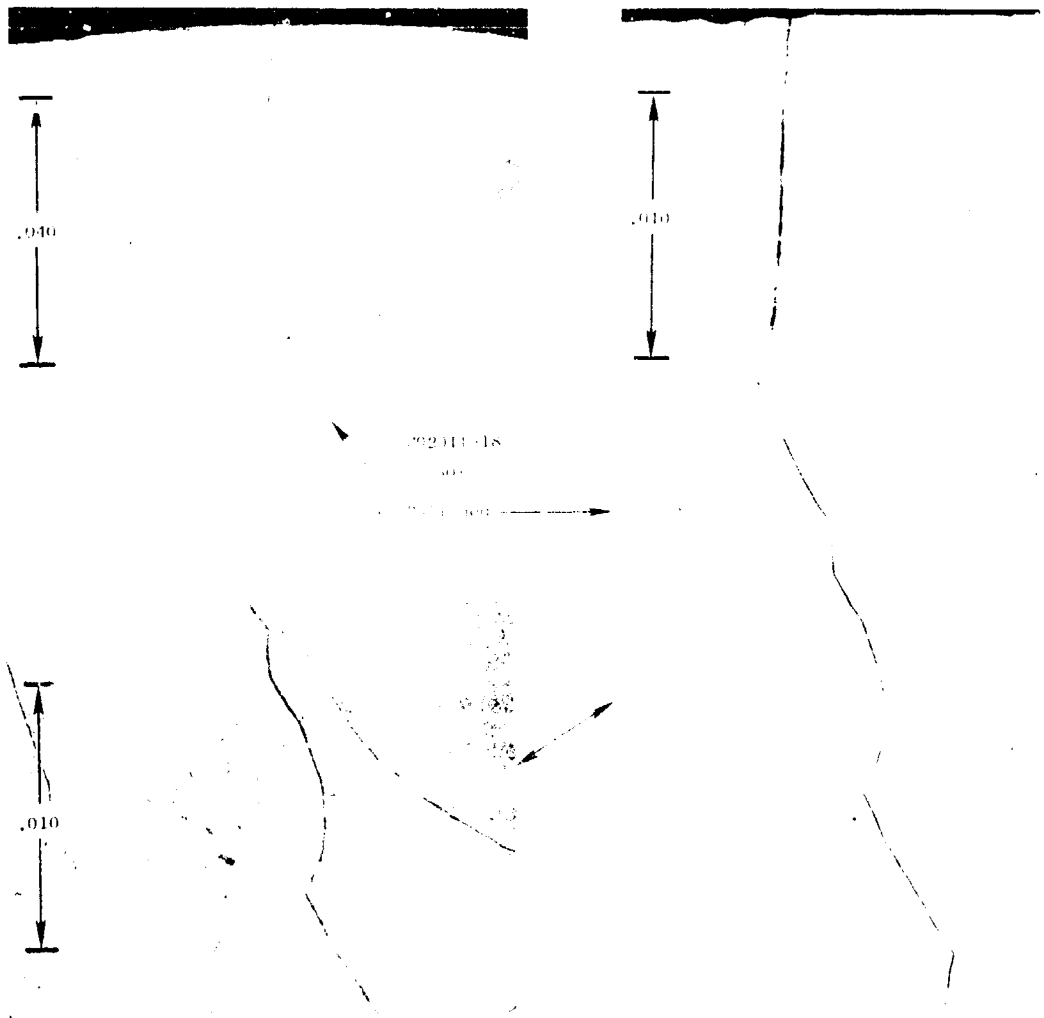
The following conclusions were derived from the metallographic examination:

1. Although the cause of the crack was not evident, it was not believed to have been initiated by alkali metal corrosion.

2. The crack appeared to have been initiated at the tube OD or lithium side and was completely intergranular. Recent tests have indicated that slight straining of T-111 weldments while hot (above 2500°F, 1370°C) results in grain-boundary sliding and separation in the weld nugget, and a small crack of this type may have been introduced during butt welding of the tube initially. A crack of this type could have propagated during bending of the tube in the fabrication of the boiler, which was performed after welding.

3. The grain boundaries in close proximity to the crack itself appeared to be influenced by the presence of lithium in the crack.

4. The favored explanation for the highly localized nature of the "apparent" lithium attack is that localized contamination of the grain boundaries occurred during the heat treatment of the boiler. This contamination could have resulted from the presence of moisture or other contaminants being present in the crack prior to the heat treatment at 2400°F (1316°C).



Etchant: 30gmNH$_4$F-20mlH$_2$O-50mlHNO$_3$

Figure K-17. Intergranular Crack in the Weld Nugget of the Boiler Tube.

8. Evaluation of the T-111 Boiler for Possible Reuse in the Corrosion Loop

The location of the leak in the boiler tube was such that a suitable repair plan could be devised; however, evaluation of the boiler material was required to document it for reuse. Specimens were removed from the boiler in the vicinity of the crack for metallographic examination and spectrographic, interstitial, and microprobe analyses. The ductility of the T-111 was determined qualitatively by compression testing.

Metallographic examination indicated no changes in the microstructure of the T-111 specimens from the boiler as a result of the alkali metal exposure and ammonia cleaning. Sections of boiler tubing were flattened without evidence of cracking or change in ductility.

The interstitial analysis results obtained on specimens removed from the boiler are compared with that obtained on as-received tubing in Table K-II. No major changes were noted as a result of the test exposure. Interstitial analysis of T-111 weld control specimens welded before and after the welding of the boiler, as part of NSP Specification No. P8AYA13-S1, "Welding of Columbium, Tantalum, and Their Alloys by the Inert Gas Tungsten Arc Process," indicated no contamination occurred during welding of the boiler.

Chemical analysis of the particulate matter found in the potassium drained from the loop indicated concentrations of Fe, Ni, Cr, and Mn. The possibility of contamination of T-111 loop materials by exposure to the particulate matter was checked by analysis for these elements. Microprobe traverses of the T-111, 0.375-inch (0.95-cm)-diameter boiler tube showed no traces (<< 1000 ppm) of Fe, Ni, Cr, or Mn. Subsequently, specimens of 0.375-inch (0.95-cm) tubing were pickled, and the acid solutions analyzed spectrographically for Fe, Ni, and Cr. These results are given in Table K-III. The higher iron and nickel concentrations observed in the first analysis of the boiler material were not observed in the subsequent analysis. Approximately 0.001-inch (0.025-mm) thickness of material was removed by each pickling operation. The slight increase in iron and nickel concentrations in the first 0.001 inch (0.025 mm) of the boiler tube wall is not believed to be detrimental.

TABLE K-II

INTERSTITIAL ANALYSIS OF 0.375-INCH (0.95-cm)-OD T-111 TUBE SPECIMENS

Element	Concentration, ppm	
	As-Received Tubing	Tubing Removed from Boiler
O[a]	36, 41[b]	40, 41
N[a]	6, 7	7, 6
H[a]	2, 1	1, 1
C[c]	37, 38	43, 62

[a] Vacuum Fusion Analysis

[b] Duplicate Analysis

[c] Combustion Conductometric Analysis

TABLE K-III

SPECTROGRAPHIC ANALYSIS OF 0.375-INCH (0.95-cm)-OD T-111 TUBING

Element	Concentration in Acid Pickle Solution,[a] ppm			
	First Pickle		Second Pickle	
	As Received Tubing	Tubing Removed From Boiler	As Received Tubing	Tubing Removed From Boiler
Fe	38	102	35	< 20
Ni	6	26	< 5	< 5
Cr	17	22	< 10	< 10

(a) Entire specimen pickled, removing 0.001-inch thickness of material from both ID and OD per pickle.

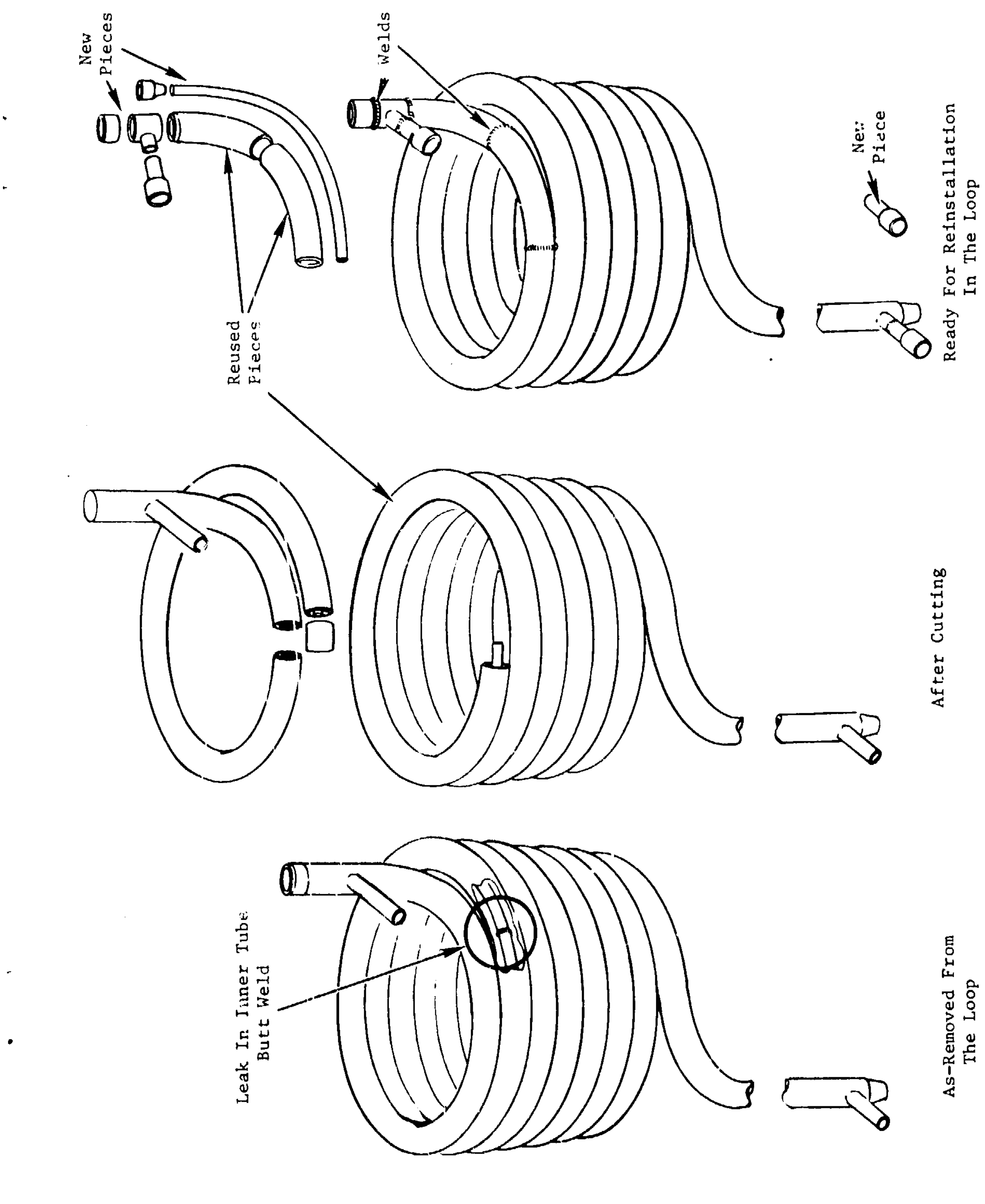
Based on the results of the various evaluations cited, repair of the boiler was recommended and drawings of the modifications needed to reuse the boiler were prepared.

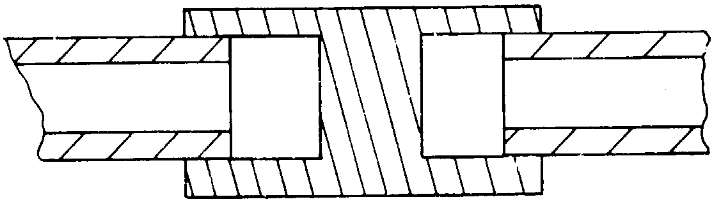
9. Boiler Repair Plan

A number of modifications to the boiler were necessitated by the repair, as shown schematically in Figure K-18. Since one coil was removed from the boiler, an additional length of T-111, 1-inch (2.54-cm)-diameter tubing was added to the top of the boiler to achieve a total boiler height equal to the original boiler for correct fitup during installation into the loop. New fittings were required for attachment to the lithium inlet and outlet lines. During installation of the boiler, the lithium lines originally on the loop were inserted into socket fittings to insure correct alignment during welding. Butt welding is normally the technique utilized for joining tubing; however, because of the location of these welds and limited access during installation of the boiler into the loop, the socket-weld approach was selected. The main concern with this type of weld joint is the possibility of an open gap between the socket fitting ID and the OD of the inserted tubing. A trial fitting was machined with a double socket, and weld experiments were performed to develop a technique to prevent this gap. The welded specimen is shown in Figure K-19. Subsequent radiographic and metallographic examination of this specimen indicated full penetration welds with no gaps were achieved when the tubing was inserted in the socket and pulled back slightly so the bottom of the tube did not contact the bottom of the socket. The joint between the 0.375-inch (0.95-cm), inner boiler tube and the 1-inch (2.54-cm), outer boiler tube at the top of the boiler was also modified with a new fitting similar to that used at the bottom of the boiler. This butt joint design is preferred over the previously employed tube-to-header joint. These design modifications are compared with the original design in Figure K-20.

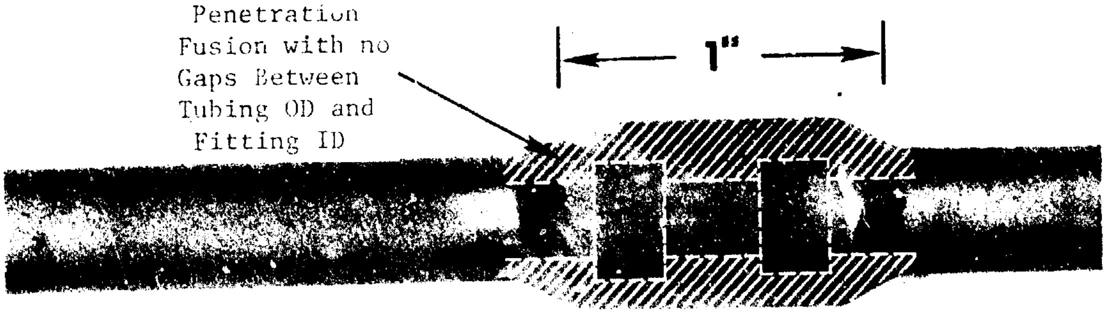
10. Boiler Repair

The necessary machined parts were received, and the boiler repair welding was initiated. Welding was performed in the VASCO welding chamber in the NSP Systems Materials Technology Laboratory according to NSP Specification No. P8AYA13-S1, "Welding of Columbium, Tantalum, and Their Alloys by the Inert Gas Tungsten Arc Process."


Leak In Inner Tube
Butt Weld
As-Removed From
The Loop
After Cutting
Reused
Pieces
New
Pieces
Welds
New
Piece
Ready For Reinstallation
In The Loop



Before Welding



After Welding

Figure K-19. Socket Weld Fitting Specimen to Qualify This Joint Design for the Boiler Repair and Reinstallation. (Orig. C68082846)

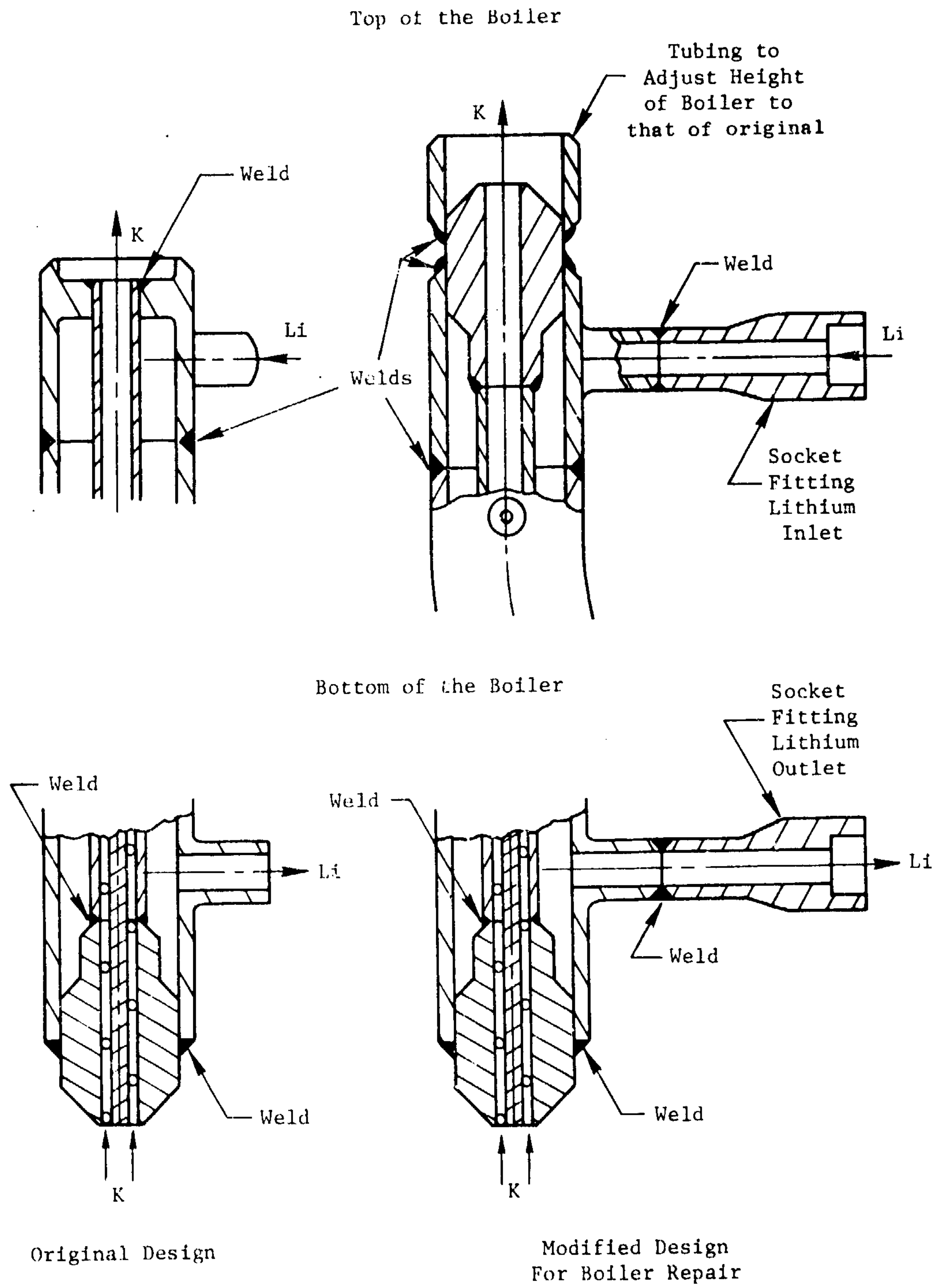


Figure K-20. Joint Design at the Ends of the Boiler.

In the initial welding step, a new section of 0.375-inch (0.95-cm)-diameter tubing was butt welded to the inner boiler tube. Subsequent helium mass spectrometer leak checking and radiographic inspection indicated this weld to be sound. Two sections of 1-inch (2.54-cm)-diameter boiler tube were then welded in place as shown in Figure K-21. These pieces were obtained from the boiler coil which was removed during sectioning of the boiler (Figure K-18). Following the joining of the new end fitting to the 0.375-inch (0.95-cm)-diameter tubing, the three welds were inspected by radiography and helium mass spectrometer leak checking and found to be sound. The boiler was completed with the addition of the lithium inlet fitting and boiler extension piece at the top of the boiler as shown in Figure K-22, and the addition of the lithium outlet fitting at the bottom of the boiler as shown in Figure K-23. The boiler plug was reattached at this time by tack welding to the bottom boiler fitting.

The repaired boiler is compared with the original boiler in Figure K-24. The removal of one coil from the boiler reduced its total length by approximately 27 inches (68.6 cm); however, the performance of the repaired boiler should not be affected since the overall length of the boiler is still greater than the length required to obtain the specified superheat (100^{o}F, 55.6^{o}C).

A final helium mass spectrometer leak check was performed on the boiler to insure no leaks were present between the potassium and lithium circuits. No leak indications were observed.

11. Postweld Annealing of the Boiler

The repaired boiler was wrapped with Cb-1Zr foil as shown in Figure K-25 and postweld-annealed according to NSP Specification 03-0037-00-A, "Postweld Vacuum Annealing of Cb-1Zr and T-111 Alloys." The chamber pressure was maintained below 1 x 10^{-5} torr (10^{-3} N/m^2) at temperatures above 1000^{o}F (538^{o}C), and the maximum pressure with the boiler at 2400^{o}F (1316^{o}C) was only 5 x 10^{-6} torr (7 x 10^{-4} N/m^2).

The boiler was again helium mass spectrometer leak checked, including across the circuits, with no indications observed.

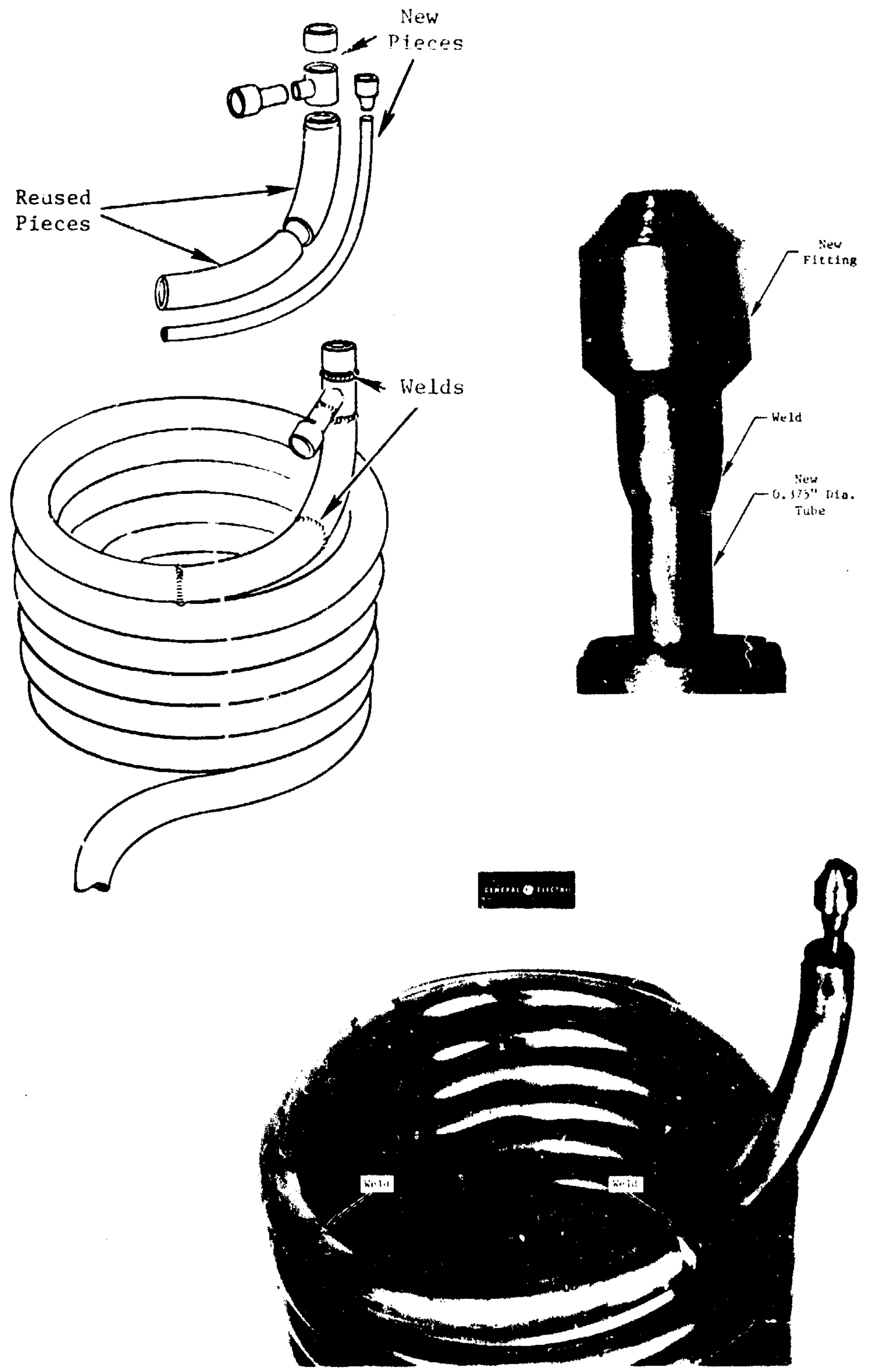
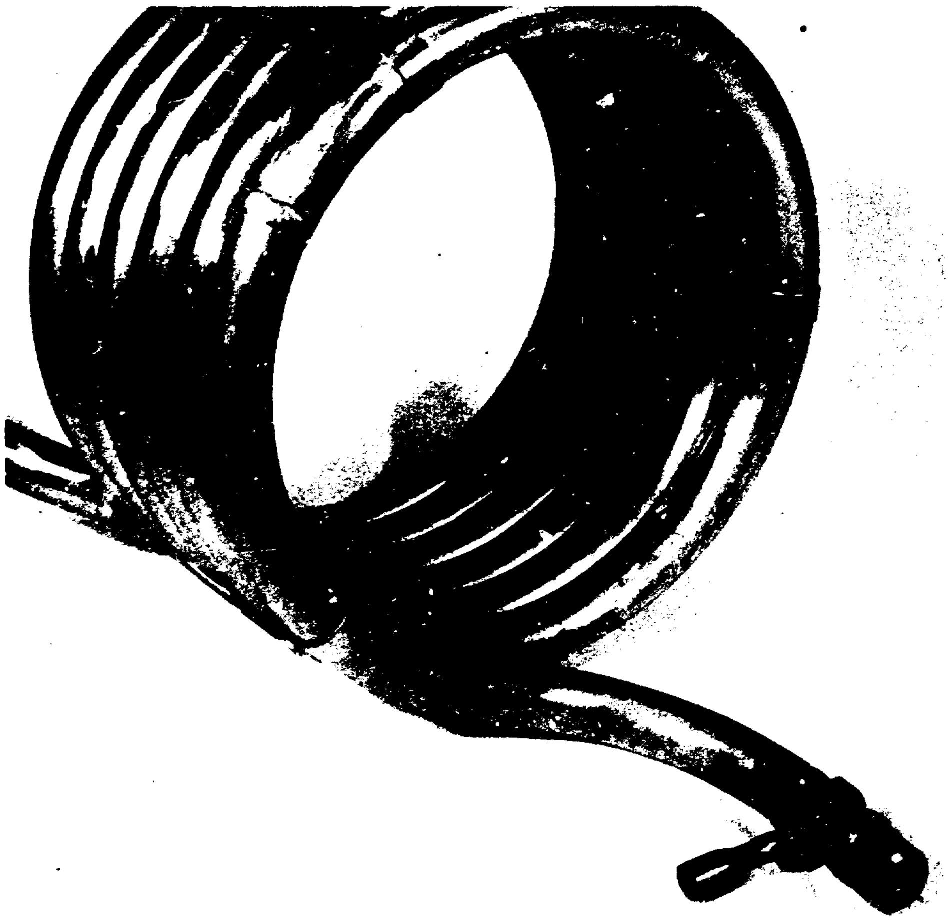


Figure K-21. Top of the Boiler Following Initial Repair Welds.
(Orig. P68-9-3A, Inset P68-9-3B)



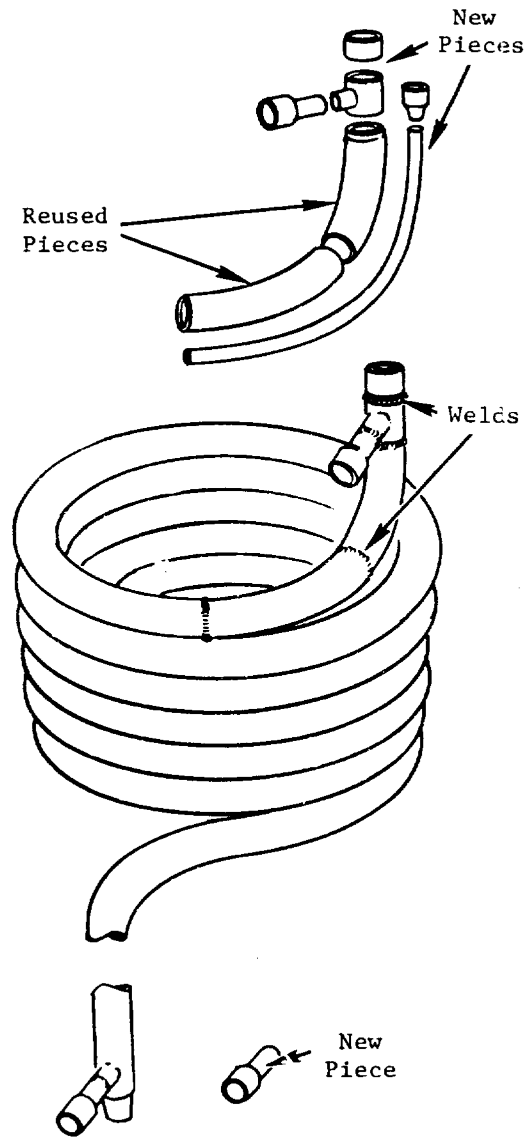


Figure K-22. Top of the Boiler Following Final Repair Welding.
(Orig. P68-9-31B)

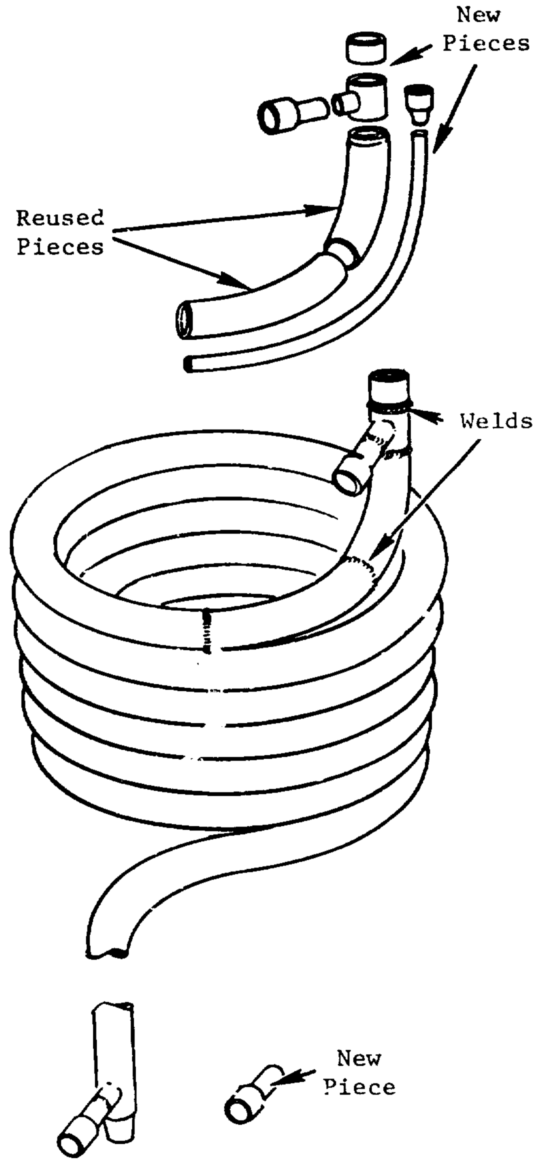
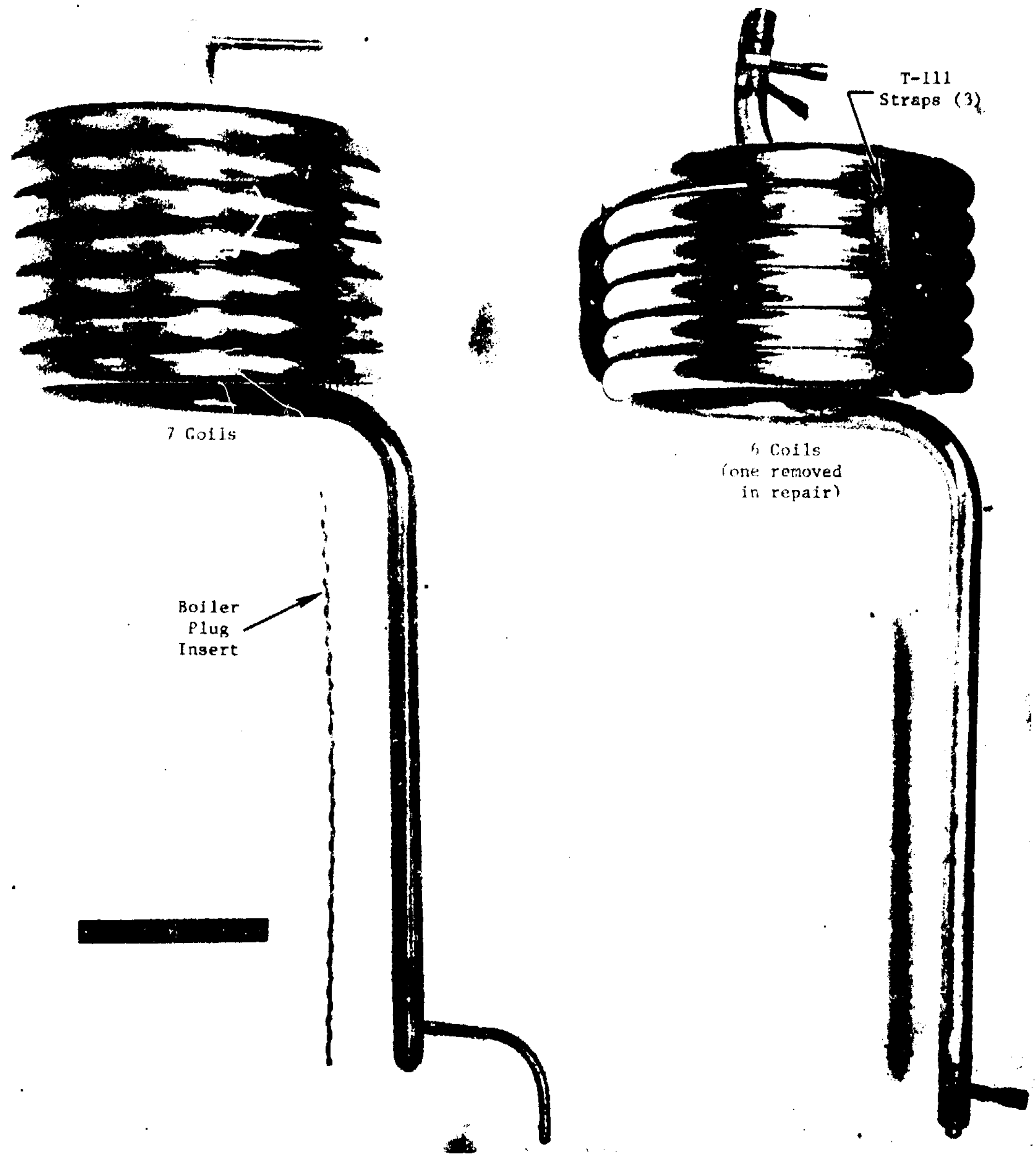


Figure K-23. Bottom of the Boiler Following Repair Welding. (Orig. P68-9-31C)



Following Initial Fabrication

Following Repair

Figure K-24. T-111 Corrosion Loop Boiler. (Orig. C67071832, P68-9-12H)

Figure K-25. Repaired T-111 Corrosion Loop Boiler Wrapped with Cb-1Zr Foil in Preparation for Postweld Annealing. (C68090903)

12. Welding Chamber for the Installation of the Boiler into the Loop

Requirements to reinstall the boiler into the loop according to NSP Specification 03-0025-00-A, "Welding of Columbium, Tantalum, and Their Alloys by the Inert Gas Tungsten Arc Process," necessitated the purchase of a special welding chamber to be installed around the loop for the welding operations. The stainless steel welding chamber was purchased from Vacuum Industries Inc., Somerville, Massachusetts. The chamber, shown during installation on the T-111 Corrosion Loop Test facility in Figures K-26 and K-27, is comprised of two flanged spool sections, four feet (1.2 m) in diameter and four feet (1.2 m) high, such that each can be rotated independently for improved access to weld locations. Sight ports and glove ports are appropriately positioned in the areas where welding will be performed at the top and bottom of the boiler location. An independently pumped tool port is also provided such that necessary tools could be brought into the chamber without contaminating the chamber environment.

The chamber is shown in Figure K-28 after final installation. Rough pumping was accomplished with the 260-liter-per-second turbomolecular pump, and the loop facility ion pumps were used to achieve the high vacuum, $< 1 \times 10^{-5}$ torr (10^{-3} N/m^2) called for in the welding specification. The chamber was backfilled with ultrahigh-purity helium which was passed through a molecular sieve dryer before entering the chamber. The inert gas analysis equipment included C.E.C.* and Panametrics** moisture monitors and the gas chromatograph shown in Figure K-29. Gas lines attached to the gas chromatograph made possible the analysis of the inlet gas as well as outlet gas from the chamber.

Before welding of the boiler was initiated, the chamber and welding equipment were qualified in accordance with the welding specification cited above.

13. Installation of the Boiler into the Loop

The boiler was welded into the loop as shown in Figure K-30. Four welds were required to reinstall the boiler; two at the top of the boiler

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* Model 26-303, Consolidated Electrodynamics Corp., Cleveland, Ohio.

** Model 1000, Panametrics Inc., Waltham, Massachusetts.

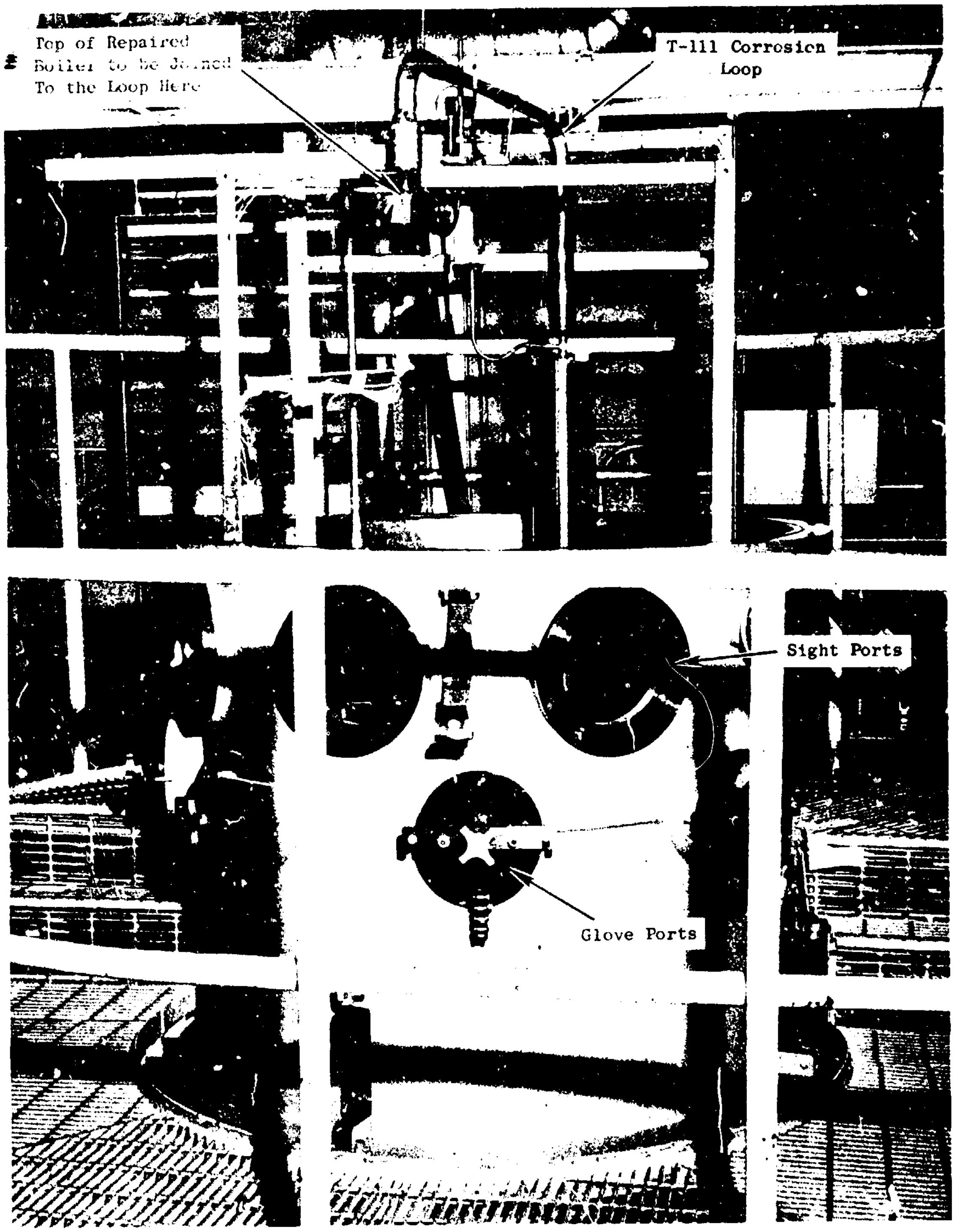

Figure K-26. Installation of the Welding Chamber Around the T-111 Corrosion Loop. Lower Spool Piece in Position. (C68090429)

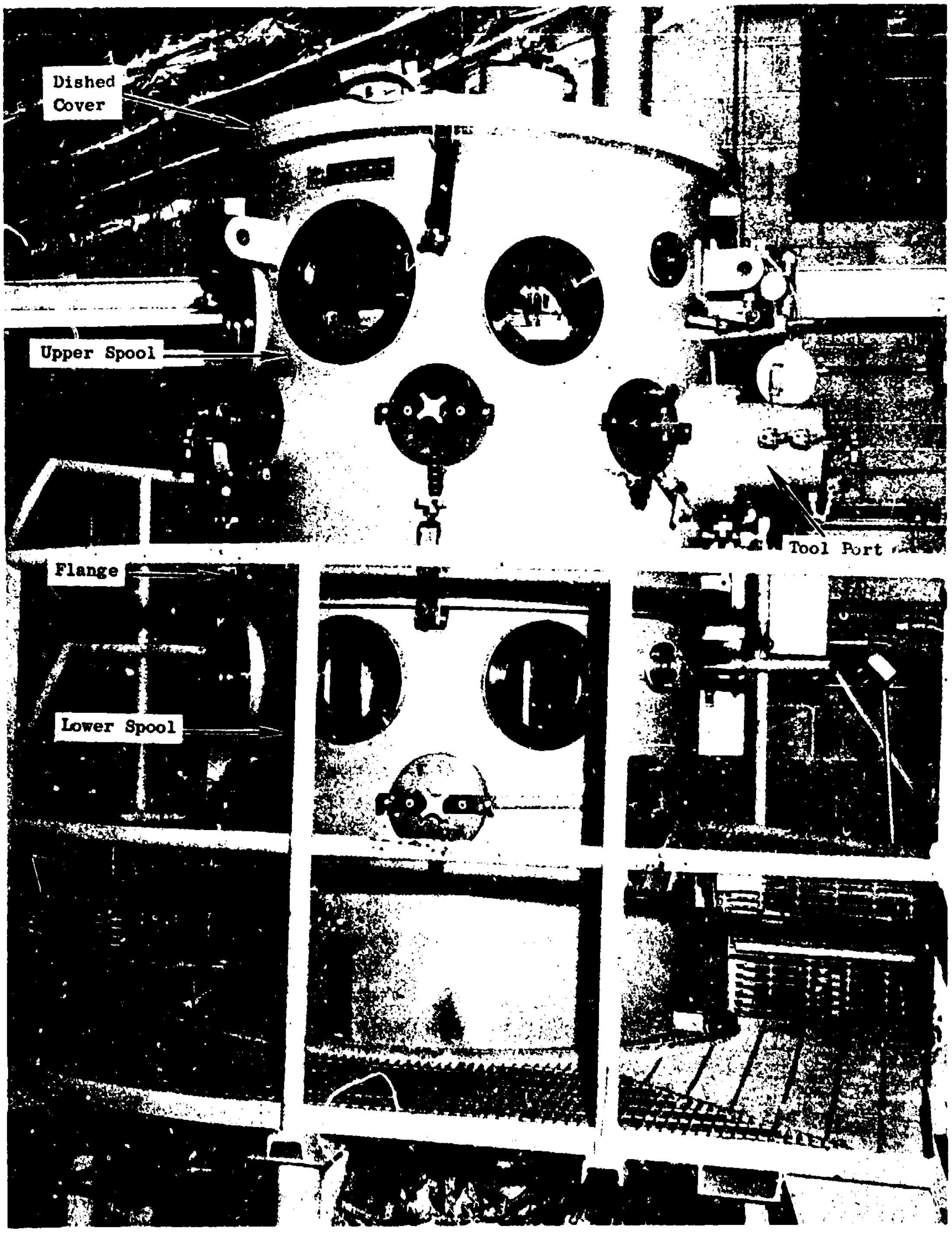

Figure K-27. Welding Chamber Installed Around the T-111 Corrosion Loop. The Chamber Consists of Two Spools Which Can Be Rotated Independently for Improved Access to Weld Locations. (C68090424)

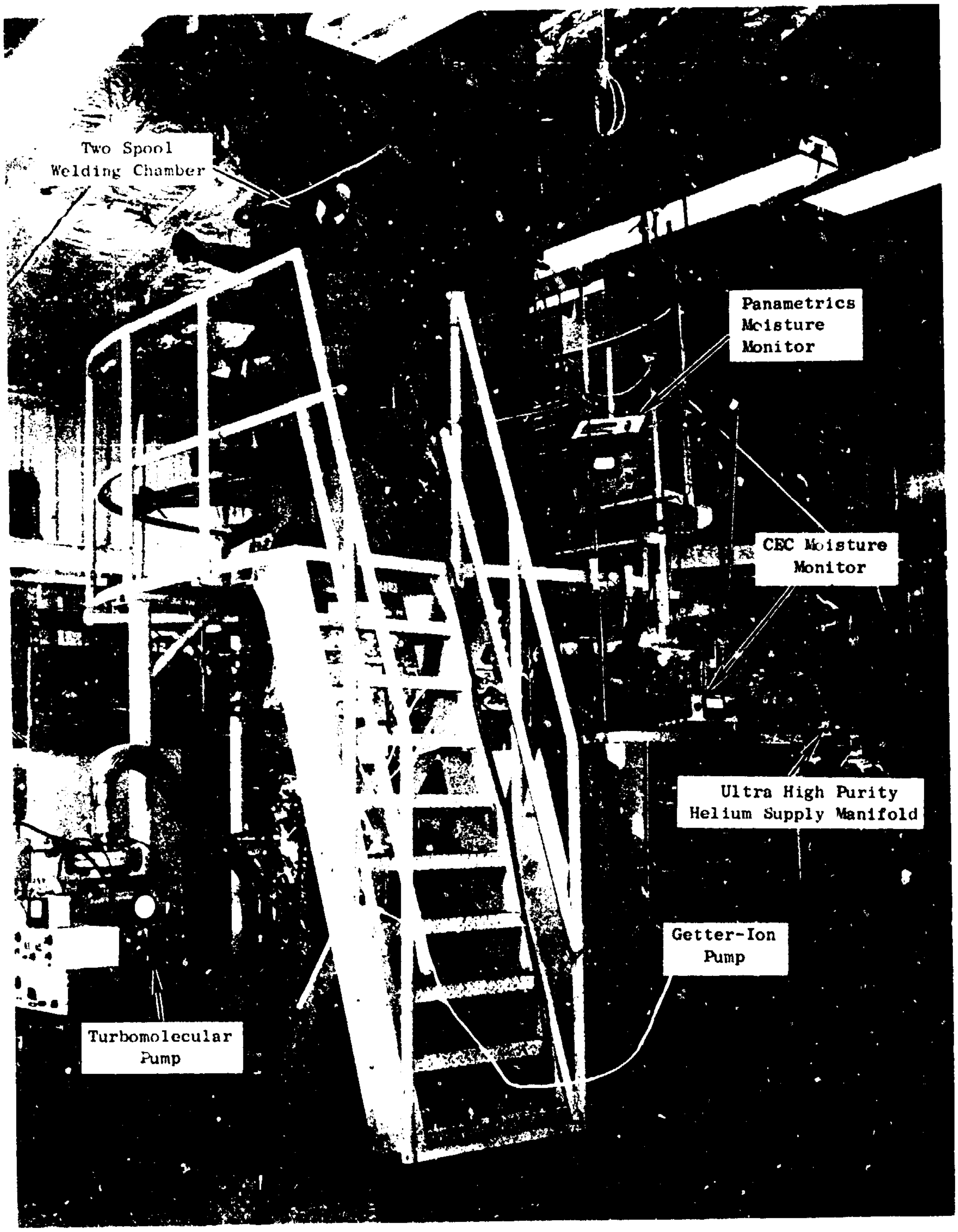

Figure K-28. Welding Chamber and Ancillary Instrumentation Installed on the Test Facility. (Orig. P68-9-44C)

Figure K-29. Gas Chromatograph Used to Analyze the Helium Atmosphere in the Welding Chamber for the Repair of the T-111 Corrosion Loop. (P68-9-44B)

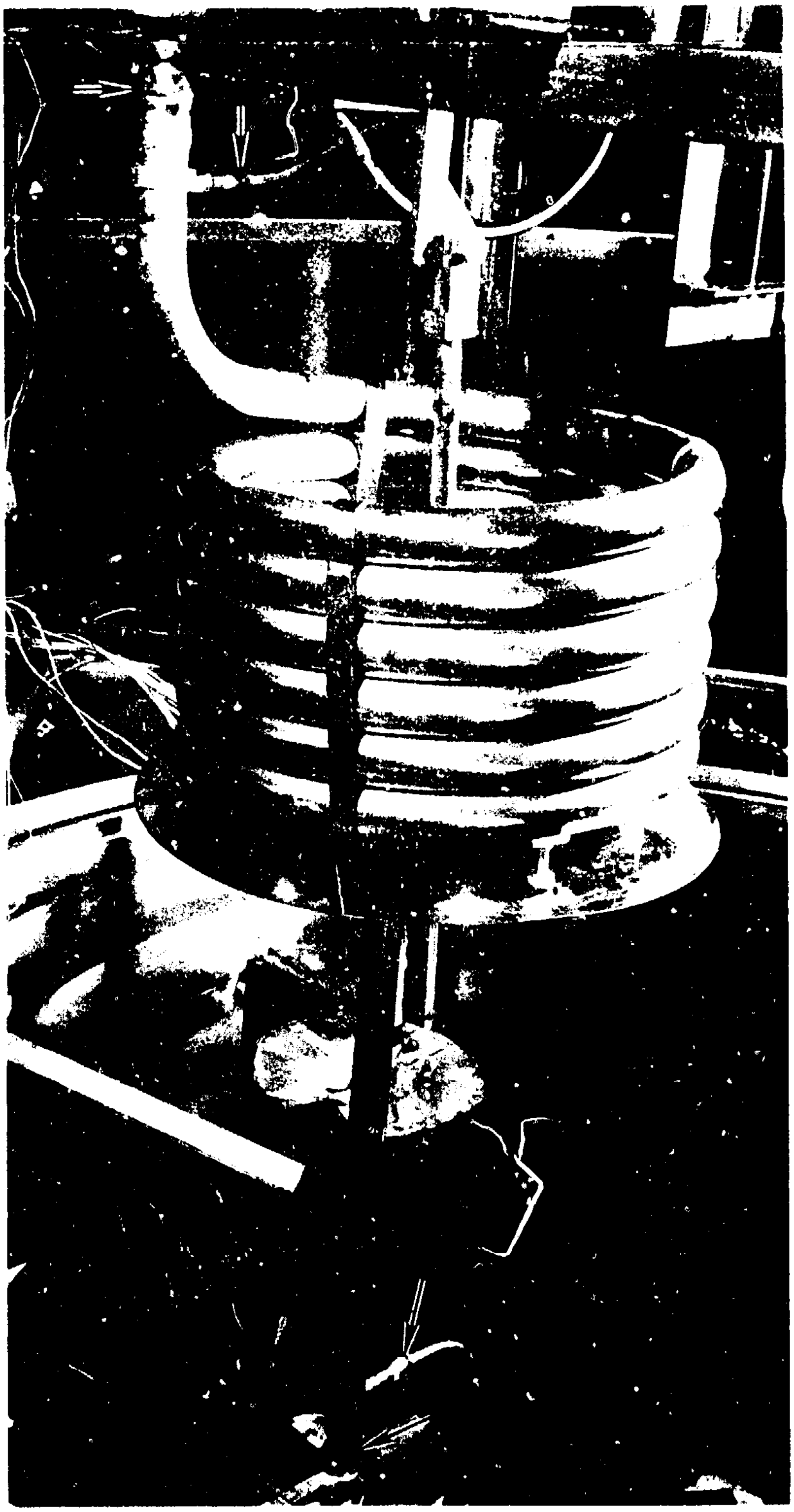

Figure K-30. Repaired Boiler Installed into the T-111 Corrosion Loop. Arrows Indicate Installation Welds. (P68-10-37B) and two at the bottom as shown in Figures K-31 and K-32, respectively. Subsequent radiographs of the welds indicated a very small area of incomplete penetration in the upper lithium line weld. Although the weld was acceptable from a joining standpoint, a decision was made to reweld the joint to improve reliability. The chamber was reassembled on the loop facility and welding completed. Subsequent mass spectrometer helium leak checking and radiography of the welds showed no indications of leaks or weld defects. The upper spool piece of the chamber was removed, and installation of the annealing furnaces around the welds was initiated. The welds were annealed for 1 hour at 2400°F (1316°C). The chamber was opened and subsequent mass spectrometer leak checking performed. No indications were noted, including a leak check between the potassium and lithium circuits.

14. Reinstrumentation of the Boiler

Reinstrumentation and reinsulation of the boiler commenced following leak checking of the loop after postweld annealing of the installation welds. Over thirty W-3Re/W-25Re thermocouples required replacement. The techniques employed for thermocouple installation and insulation with Cb-1Zr dimpled foil were described in Section VII.

15. Test Facility Operations

Following reinstrumentation and reinsulation, the chamber was closed and evacuated with the turbomolecular pump. Mass spectrometer leak checking was performed, and no leaks were found. Ion pumping was initiated, and the bakeout heaters were then turned on. At that time, a final helium check was performed between the potassium and lithium circuits with the loop at 375°F (190°C), and no leaks were found. Bakeout continued until alkali metal flushing of the loop circuits was completed.

16. Alkali Metal Flushing of the Loop

As described previously, examination of the alkali metals drained from the loop indicated lithium in the potassium and potassium in the lithium as a result of the boiler leak. Particulate matter was also found in the potassium. The particles and contaminated alkali metals were removed from the loop by repeatedly flushing the circuits with pure alkali

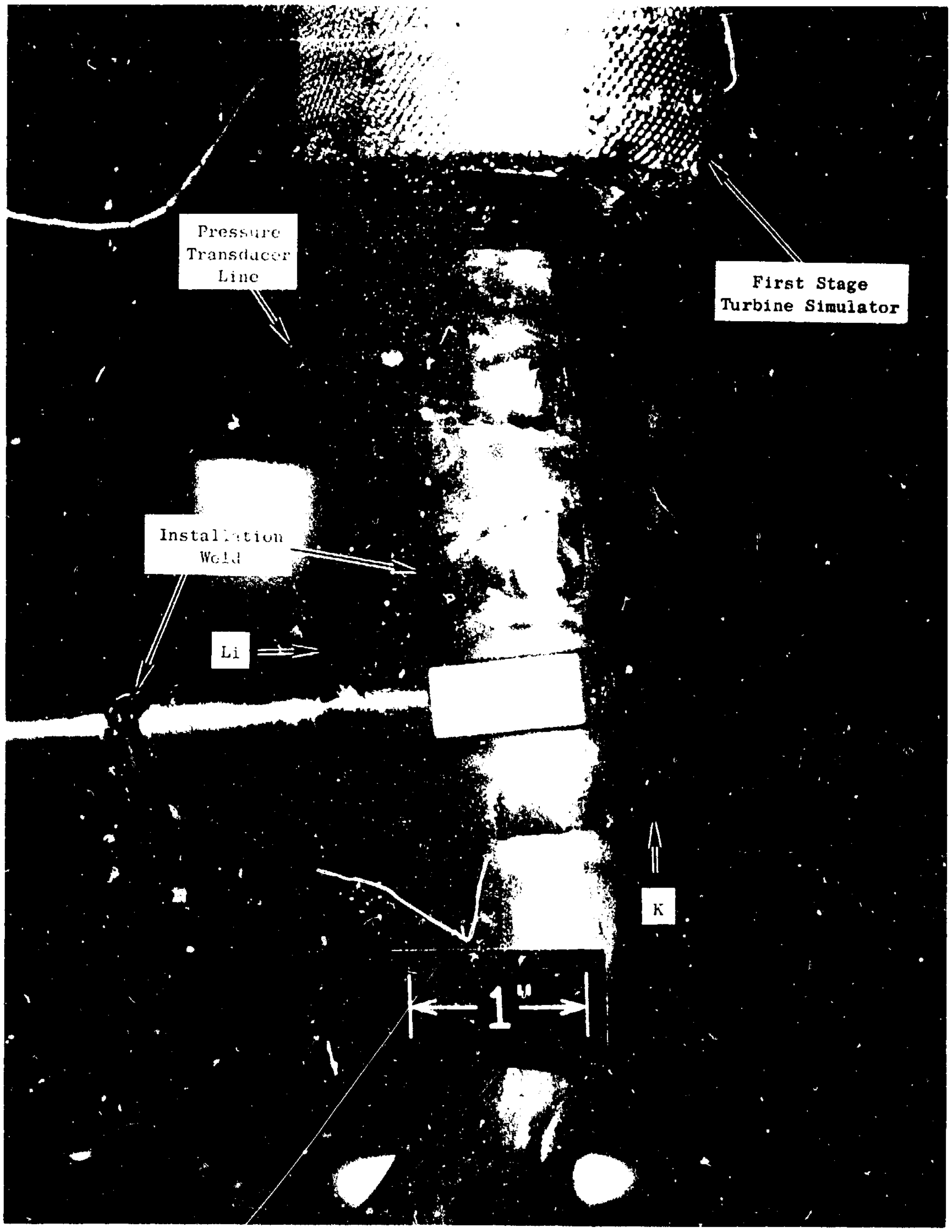

Figure K-31. Top of Repaired Boiler After Installation in the T-111 Corrosion Loop. (P68-10-37A)

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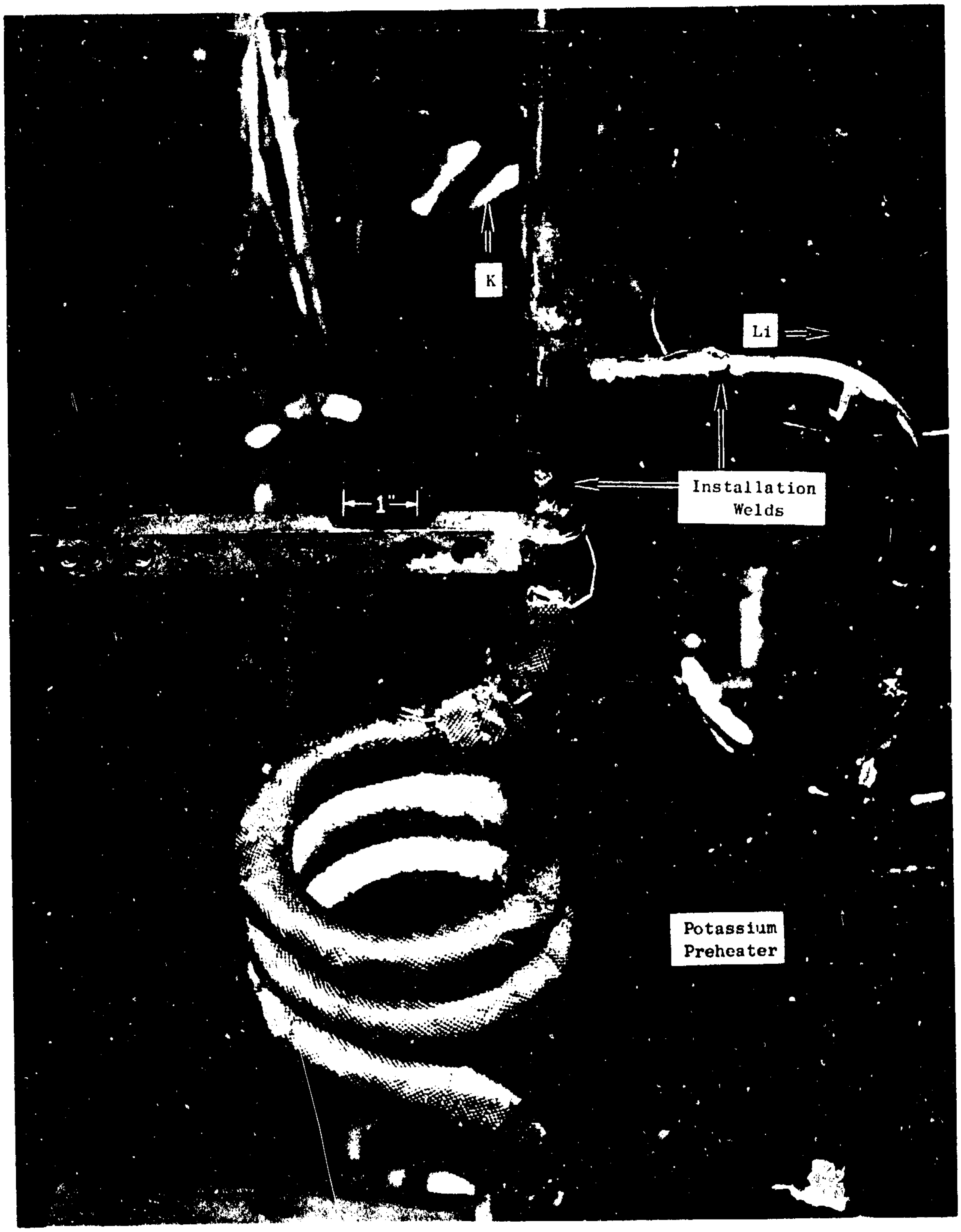

Figure K-32. Bottom of Repaired Boiler After Installation in the T-111 Corrosion Loop. (P68-10-37C) metals. Since no useful data could be found in the literature regarding the material solubility of potassium and lithium, a limited solubility study was performed to obtain data which would be useful in determining the adequacy of successive flushing operations in removing residual lithium from the potassium circuit and in removing residual potassium from the lithium circuit. The results of these tests are given in Appendix L, Lithium-Potassium Solubility Study.

The apparatus required for filling and flushing the potassium loop circuit is shown in Figure K-33. The transfer system was modified for flushing operations by inserting a dump line between valves FF, KK, and the disposal tank. This permitted the transfer of flush charges of potassium directly to the disposal tank without contaminating the fill system upstream from valve FF. In addition, the dump line was connected to a small potassium still where flush potassium charges could be diverted for subsequent distillation and analysis of the residue for lithium concentration and particulate matter.

Subsequently, the potassium side of the transfer system was filled, and the potassium was dumped into the disposal tank to flush the system. The system was refilled with potassium and a sample obtained. The analysis of this sample, shown in Table K-IV, was acceptable, and the potassium surge tank was filled with an 1800-cc charge to initiate flushing operations.

The secondary circuit was flushed with four 1800-cc charges of potassium while the final purification of lithium was in progress. The various regions of the loop were maintained at temperatures in the 700^{o}F (371^{o}C) to 1050^{o}F (566^{o}C) range during the flushing operations.

The first charge was used to establish circulation and, after the flow was reversed to agitate particles, was quickly dumped into the disposal tank. Flow indications during circulation indicated some plugging of the metering valve with particles, and the valve wa actuated to its full open position. Improved flow indications were obtained during the circulations of the second and third potassium charges with some observed decrease in the pressure drop across the valve. Both the second and third charges were circulated four times before removal from the surge tank. The second charge was dumped into the disposal tank. The third charge was

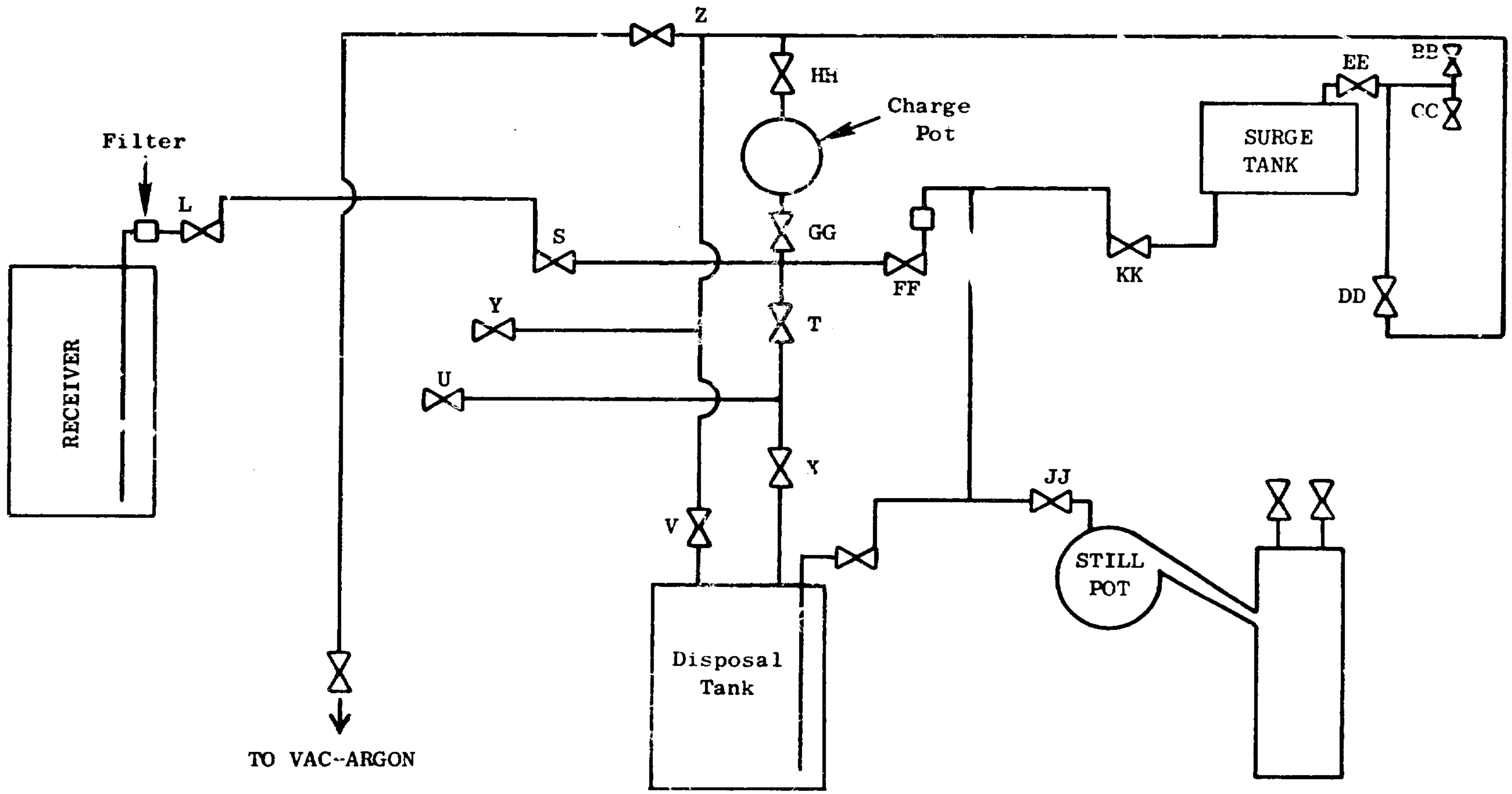

Figure K-33. Potassium Transfer System for Filling and Flushing the T-111 Corrosion Test Loop.

TABLE K-IV

ANALYSIS OF THE POTASSIUM USED IN FLUSHING THE T-111 CORROSION TEST LOOP[(a)]

Element	Concentration, ppm
O	7,14
C	51
Ag	< 2
Al	< 2
B	< 30
Ba	< 10
Be	< 2
Ca	< 2
Cb	< 10
Co	< 2
Cr	< 2
Cu	< 2
Fe	< 2
Mg	< 2
Mn	< 2
Mo	< 2
Na	< 20
Ni	< 2
Pb	< 20
Si	< 2
Sn	< 20
Sr	< 2
Ti	< 10
V	< 20
Zr	< 10

(a) Analysis obtained on a sample removed from the transfer system before transferring the potassium into the loop surge tank.

dumped into the still for subsequent analysis. The potassium was distilled off and the 39 mg of residue analyzed for lithium. A concentration of approximately 30 ppm lithium in the potassium was determined. A very small amount of particulate matter was found in the still. The still was cleaned, welded together, and reinstalled in the transfer system as shown previously in Figure K-33.

The lithium transfer system was subsequently flushed by filling and draining the lithium into the disposal tank. The transfer system was refilled and the lithium sampled. The analysis, presented in Tabke K-V, was acceptable, and the lithium surge tank was filled.

The first lithium charge was discarded into the disposal tank, and the surge tank was refilled with a 1600-cc charge of lithium. The lithium was circulated in the primary circuit and used to heat a fourth charge of potassium to higher temperature than previously possible with only potassium in the loop. The potassium charge was circulated at temperatures of 530°F (277°C) to 1420°F (771°C) and subsequently dumped into the surge tank three times.

The potassium was distilled off and the 28-mg residue analyzed for lithium. A concentration of approximately 20 ppm lithium in the potassium was determined. No particulate matter was found in the still. The data indicated sufficient flushing of the potassium circuit had been performed.

The lithium charge in the primary circuit was dumped into the disposal tank and the loop filled with a third charge of lithium (1600 cc). The lithium sampler was installed at valve KK and, after circulating the lithium in the primary, a sample was taken. Analytical results indicated a concentration of 90 ppm potassium in the lithium. Since the solubility limit for potassium in lithium at the sampling temperature (580°F, 304°C) was 1000 ppm, and the potassium concentration in the distilled lithium was 55 ppm, further flushing of the lithium primary circuit was believed unnecessary.

17. Filling the Loop with Alkali Metals for Operation

The secondary loop was filled with 2300 cc of potassium, and the primary loop was filled with 2200 cc of lithium. Samples were withdrawn from each loop for final qualification analyses. The analytical results, shown in Table K-VI, indicated acceptable purities, and circulation of the alkali metals was initiated.

TABLE K-V

ANALYSIS OF THE LITHIUM USED IN FLUSHING THE T-111 CORROSION TEST LOOP[a]

Element	Concentration, ppm
N	34
O	20
C	46
Ag	< 5
Al	25
B	< 50
Ba	< 50
Be	< 5
Bi	< 25
Ca	25
Cb	< 25
Co	< 5
Cr	< 5
Cu	5
Fe	< 5
Mg	5
Mn	< 5
Mo	5
Na	< 50
Ni	5
Pb	< 50
Si	25
Sn	< 25
Sr	25
Ti	< 25
V	< 25
Zr	< 25

(a) Analysis obtained on a sample removed from the still receiver following hot trapping and vacuum distilling.

TABLE K-VI

ANALYSIS OF THE ALKALI METALS USED IN OPERATION OF THE T-111 CORROSION TEST LOOP[(a)]

Element	Concentration, ppm	
	Lithium	Potassium
N	35,39,46,49	-
O	43	3,6
C	31	32
Ag	< 5	< 2
Al	5	10
B	< 50	< 30
Ba	< 50	< 20
Be	< 5	< 2
Ca	5	10
Cb	< 25	< 10
Co	< 5	< 2
Cr	< 5	< 2
Cu	5	2
Fe	< 5	2
Mg	5	2
Mn	< 5	< 2
Mo	< 5	< 2
Na	< 50	< 20
Ni	5	< 2
Pb	< 50	< 20
Si	5	2
Sn	< 25	< 10
Sr	5	< 2
Ti	< 25	< 10
V	< 25	< 10
Zr	< 25	< 10
Li	-	31
K	106	-

(a) Analysis obtained on samples removed from the loop prior to initiation of loop start-up.

APPENDIX L

LITHIUM - POTASSIUM SOLUBILITY STUDY

Communication between the two circuits of the loop resulting from the leak in the potassium containment tube in the boiler caused potassium contamination of the lithium circuit and vice versa. Successive flushing of the two circuits with alkali metal was considered to be the most logical method of removing this contamination; however, no useful data was available in the literature regarding the mutual solubility of lithium and potassium. Therefore, an investigation was conducted to determine this information during the period during which the boiler was being repaired. This data and the methods used to obtain it are discussed below.

A schematic diagram of the solubility apparatus is shown in Figure L-1. The apparatus was filled with equal volumes of lithium and potassium which had been previously purified by vacuum distillation. The apparatus was brought to a specific temperature and held for at least 24 hours to allow equilibration of the alkali metal solutions. Samples of lithium and potassium were taken at the temperature of interest. The entire sample was analyzed to determine the total lithium concentration in potassium or potassium in lithium since the solubility will change during cooling of the sample. The special sampling system used in this study is shown in Figure L-2.

The mutual solubilities were determined over the 600°F (316°C) to 1200°F (649°C) temperature range in the apparatus described. The data obtained are presented in Table L-I. These data were obtained by equilibrating equal volumes of lithium and potassium at constant temperature for at least 16 hours. The data shown for 800°F (427°C) represent times of 16 hours and 24 hours with no change in solubility indicated. No data was obtained for the solubility of lithium in potassium at 1200°F (649°C) because the valves used did not function properly after prolonged exposure to this temperature. The temperatures were measured with calibrated chromel/alumel thermocouples accurate to ± 10°F.

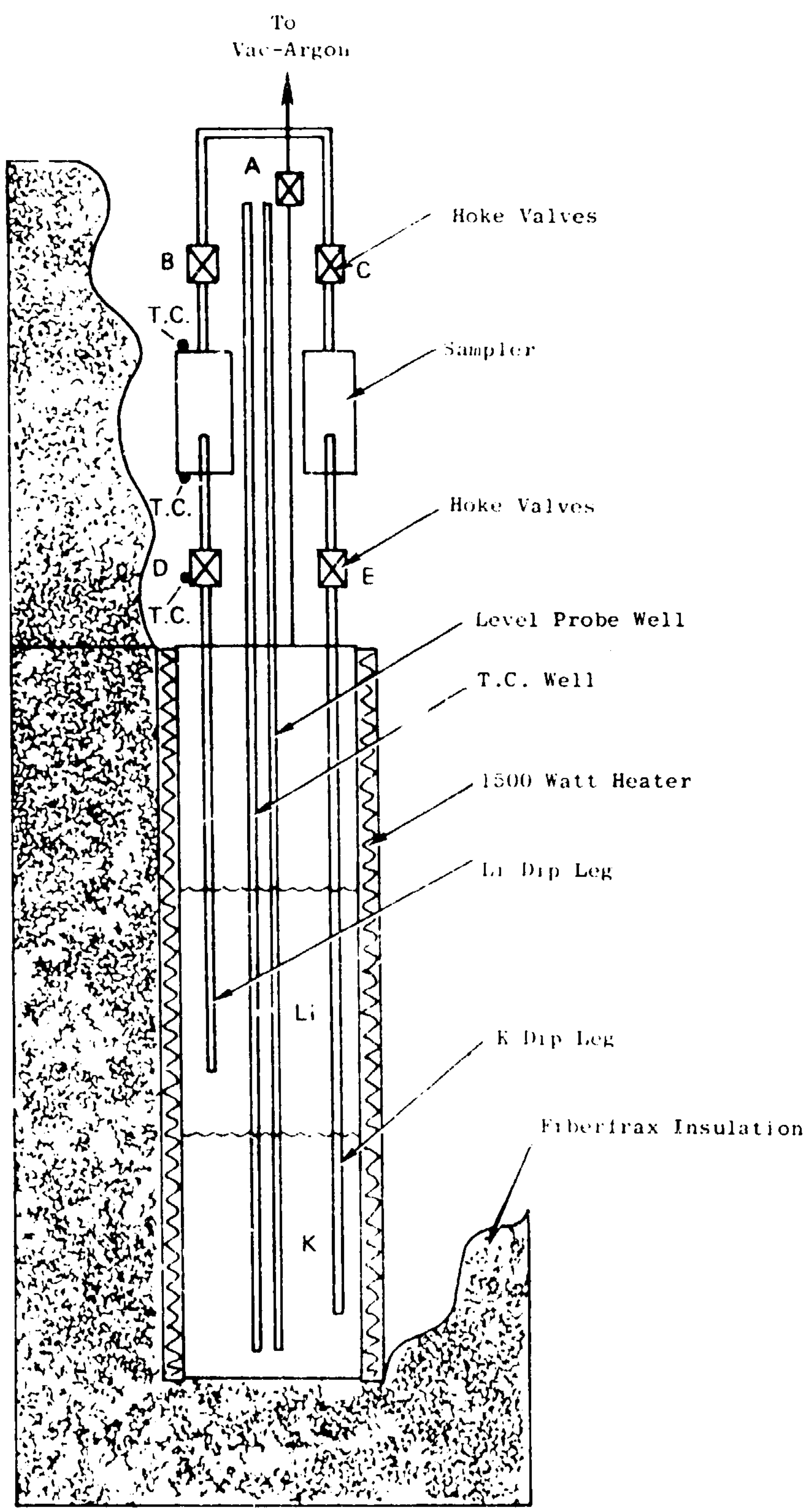

Figure L.1. Lithium-Potassium Solubility Apparatus.

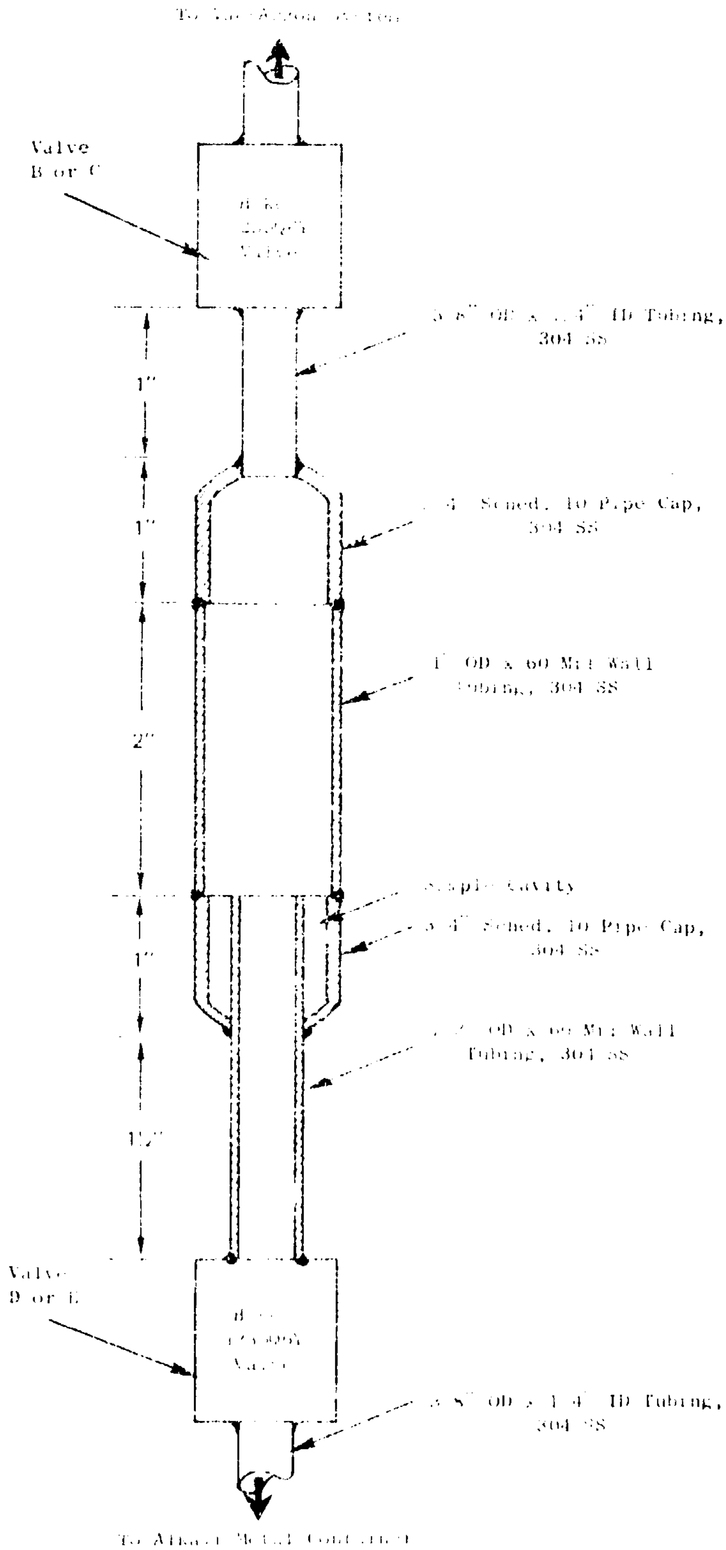

Figure 1-2. Lithium Potassium Solubility Study Sampler.

TABLE L-I

MUTUAL SOLUBILITIES OF POTASSIUM AND LITHIUM

Temperature °F	Temperature °C	Wt. % K[a] in Li	Wt. % Li[a] in K	Run No.
610	321	0.10	0.23	4
610	321	(b)	0.23	5
800	427	0.19	0.36	1
800	427	(b)	0.37	2
1000	538	0.70	1.12	3
1230	666	2.60	-	6

(a) Determined by spectrophotometric analysis.

(b) Sample lost in preparation.

U.S. Government Printing Office: 1976 660-086/23 Region 5-11